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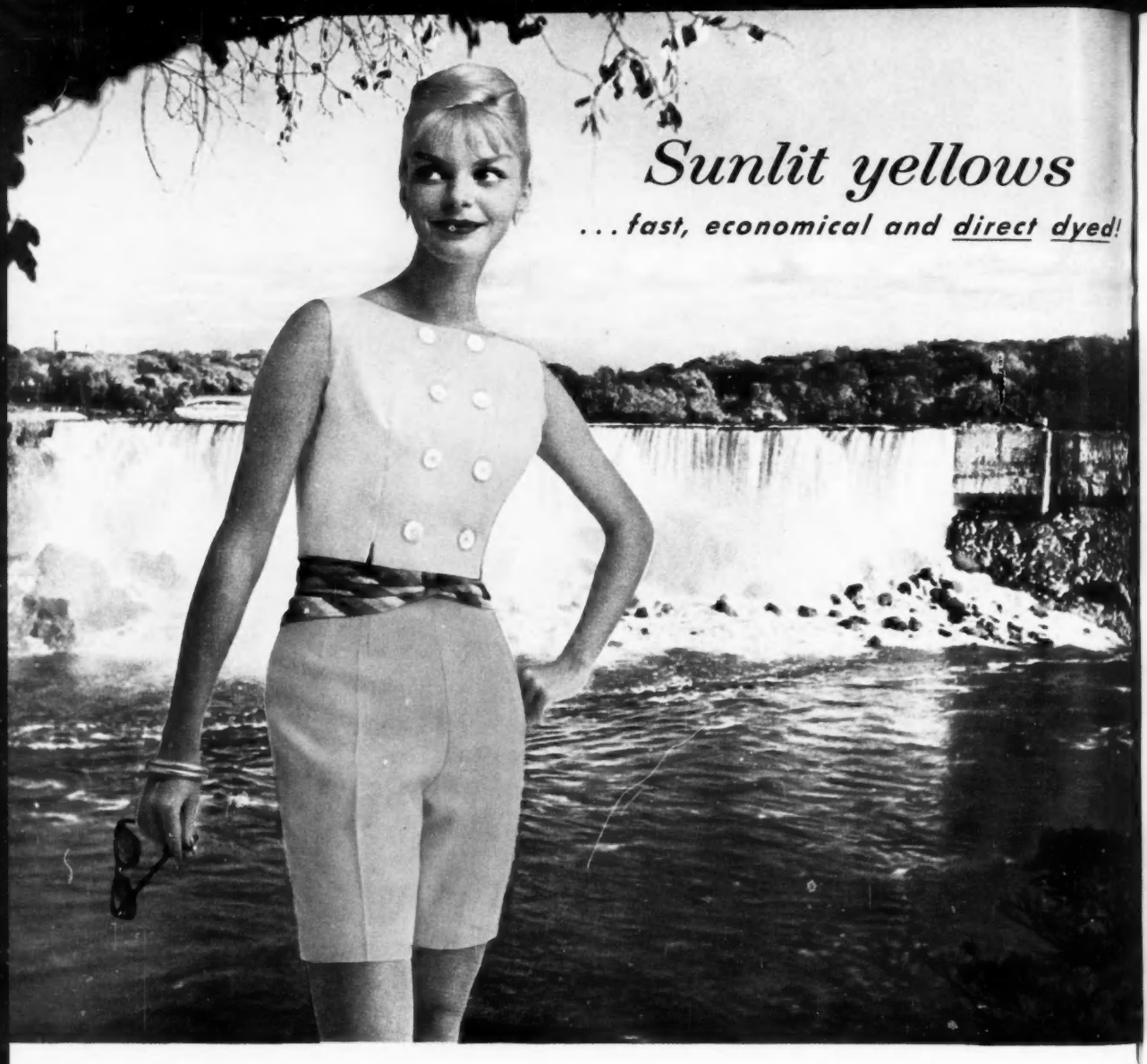

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AMERICAN

DYESTUFF REPORTER

Volume 49 February 8, 1960 Number 3

NEW ARTIFICIAL WEATHERING METHODS
Rubber, Plastics and Textiles*

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INTRODUCTION

THE general purpose of this paper is to present briefly the problems and progress (including a portion of the author's new data) mainly since 1955 in the artificial weathering of materials in general in the United States and abroad. Emphasis is on broad fundamentals, but with specific examples when necessary.

Artificial weathering depends obviously on the best possible reproduction of all major causes of the weathering effect for some specific area. While this simulation is admittedly a somewhat difficult problem, notable progress toward solutions has been made in recent years in the United States and abroad, but unfortunately, not always jointly. If the international character of this paper reduces this divergence, the author will be gratified.

ARTIFICIAL WEATHERING: OBJECTIVES; CAUSES AND EFFECTS; LIMITATIONS

OBJECTIVES——The primary ideal objective of artificial weathering (see definitions in Appendix I) is the exact simulation of the natural weathering of a specific area. While this is practically impossible because of the many variables involved at only one area, some standard testing parameters can be established (*1,2,3*). Then the advantages are: a) products of better serviceability during their lifetime; b) exposure time will be decreased; c) reliable comparisons of exposed materials may be made using approved standard variables; d) products are improved in service, behavior and acceptance tests; and e) new knowledge may be acquired.

Important topics included in this paper are as follows: effects of sunlight, moisture (including rain), temperature, gases, nuclear radiation, dust and wind; and instrumentation for controlling some of these quantities at preset values in the test space.

CAUSES——To simulate the natural weathering of many kinds of materials requires: a) a knowledge of the important causes of the weathering effect, and b) a suitable substitute for these causes in terms of their controllable parameters, eg, a light source closely approximating natural sunlight at the earth's surface, as in Figure 1. Most of these causes are presently known (at least for weathering at the earth's surface (*18*). These, of course, involve natural weather's components and their action—physical and chemical—with resulting subsidiary actions, such as change in physical and appearance properties. (Biological agents, such as micro-organisms which produce effects typified by mildew, are not included here, but it should be mentioned that a preliminary ASTM committee on plastics has already provided an interim report.) The following necessarily brief description of such components and actions may be helpful.

A) *Sunlight.* As received at the surface of the earth, the solar radia-

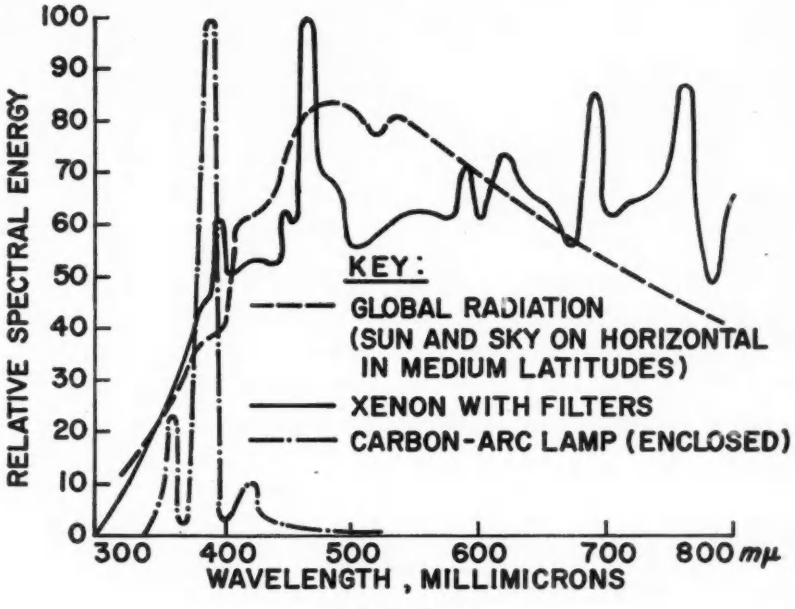

Figure 1

Comparison of relative spectral energy vs wavelength of xenon and carbon-arc light sources with natural sunlight

* Presented at the International Rubber Conference held in Washington, DC, on November 11, 1959.

tion spectrum may be divided approximately as follows: 1) ultraviolet radiation which ranges from 200 (on high mountains) to 380 mμ, about four percent of the total; 2) visible radiation which ranges from 380 to 760 mμ, about 43 percent; and 3) infrared radiation which ranges above 760 mμ, about 53 percent of the total radiation.

Intensity of July solar radiation of approximately 40° North latitude is about 700 langleys per day over most of the USA on clear days. This approximate value is used reliably in artificial weathering estimates (*56*).

Solar radiation absorbed by the material is converted into heat. That which is reflected determines the hue or color of the material. When a dye molecule, for example, absorbs UV or visible light, its resulting excited state may change or destroy it in this photo-chemical reaction. Earlier studies of such reactions from an organic chemical standpoint have recently been replaced by those from the physical and physical-chemical standpoint (*54*).

B) *Temperature.* The diurnal (daily) range of temperature varies considerably, both with season and location. It is large near the ground at high-level stations, over sand, cultivated fields and rocks; small over thick vegetation and deep water, and practically zero in free air at about 4,000 feet above the surface. Recent monthly averages in Washington, DC, ranged from about 35° Fahrenheit in December to about 77° Fahrenheit in July, but the extremes were 14 and 95° Fahrenheit. Such extremes cause severe physical and chemical degradation. Incidentally, it should be noted that chemical reactivity of a material in general increases exponentially with the temperature.

C) *Moisture.* Of its two main components, humidity and rain, humidity is greater in its overall weathering effect. It depends on the time of the day and year and can range from 10 to 100 percent. It is usually low during the warmest part of the day and high when the temperature has dropped to its minimum. Depending on the dewpoint and temperature, condensation occurs so that in warmer regions, particularly along seacoasts where the air is moist, more condensation occurs than in the cooler or drier regions (*34*). In general, humidity constitutes a moist continuum in constant contact with material to produce hydrolysis, leaching and similar effects, or accelerate dispersion of aggregates (of say, a dye) into smaller particles. Fading is greater for smaller particles (*29*) since the ratio of the area (where fading occurs) to volume varies inversely with the diameter of the particle. Millson has said (*56*) that moisture can explain many earlier anomalies in natural light fading of textiles. Perhaps his most striking example is that of a rug from a formulation whose serviceability throughout this country was very satisfactory for several years. When it was covered by a thin plastic film for several days in service it faded considerably. Lab duplication of presumably the same service conditions produced the same fading. Millson's explanation, which seems quite plausible, is that nighttime condensation from naturally moist air trapped between the rug and plastic film contributed largely to the same fading phenomenon characteristic of his other similar experiments and exhibits on water-wetted materials.

Rain can produce quite different effects because of its washing or its impacting action (*17*). The former removes foreign-deposited material and those material particles which have formed on or under the surface; leaching action is a typical example. The impacting produces a sandblasting action when the rain particles carry small particles such as dust. In erosion studies, for example, one must always consider the average drop size distribution, water content, and drop velocity. In addition to rain, one should consider also rain, hail, and sleet (*34*).

Moisture, both as vapor or rain, is more readily held, and resides longer, in a material's indentations to contribute its effect in correspondingly increased measure. For example, particle dispersion, stress relaxation, and temperature of a material would be changed by such micro-depositions of moisture.

It is said that wet materials absorb more heat than dry, which is contrary to the common assumption that they should be cooler.

D) *Wind.* Wind, which is air in motion, acts as a carrier of impurities such as dust, gases, moisture. Its speed may increase rapidly with altitude from the ground and at the ground is lowest at night and greatest during the afternoon. This is sometimes determined by geography or contour. Gas transport over long distances also contributes to the fickle nature of the weather; for example, ozone found in the stratosphere is brought to the earth's surface by the wind. The absence of wind is often detrimental since collections of sometimes lethal gas result, as in a smog area (*17,30*).

E) *Dust.* This includes all foreign matter, such as smoke, soot and salts. Its daily variation is due to temperature gradient of the surface atmosphere; at night, and in the early morning hours, the dust settles to the lowest layers. But, as the temperature rises during the day and the wind increases, it is lifted and distributed at higher levels. Its presence effects not only materials, but plants and animals (*18, 30*). In the seacoast areas, sea salt nuclei present serious corrosion problems.

F) *Gases.* The nature, quantity and distribution of gaseous impurities depends largely on the region and wind, eg, one finds in industrial areas oxides of carbon, nitrogen, sulfur and fluorine, together with their reaction products. Of the latter, smog has been a fashionable example since 1947 when experts thought its only source was the stratosphere, but it is primarily a reaction product (*30*) with harmful effects on materials and men. The action is chemical and too extensive to detail in this limited paper.

G) *Miscellaneous.*

1) *Nuclear Radiation.* This is offered as a possible explanation of some of the many "anomolous results", a catch-all term often used for either convenience, shelter or caution. As in the cases of gases, very recent and preliminary speculations indicate that some nuclear radiations may produce small weathering effects (*32,58*). The cause may originate from a variety of sources including the upper atmospheres (by either man-made or natural means) or outer space as in the case of cosmic rays. Nuclear reactor apparatus, whether used in stationary or moving surface or air installations, produces serious deteriorations of most rubbers, mainly from neutron and gamma radiation (*58*). A common example today is the effect of nuclear radiation on a photographic emulsion. It is known that (*32*) the major reactivity in polymers depends mostly on the the total energy absorbed, the radiation intensity and rarely on the type of radiation or its source. For example, in cross-linking of polyethylenes

or silicones, the degree of cross-linking depends only on the energy absorbed and not on the type or source of energy. In nonpolymers, such as water, the results would depend on the type of radiation (alpha, X-ray, etc).

2) *Electrically charged particles.* These may possibly explain anomolous results. For example, physical shielding of sunlight from a sensitive electrometer outdoors shows a marked decrease of ionization (*18*). Photoionized air from sunlight will produce positive and negative ions such as oxygen in or near a material to produce discernible physical or chemical degradation. The general action is one of accelerating chemical reactions, the products of which can be harmful to materials. Incidentally, smog irritants are the results of such sunlight - stimulated reactions (*17*).

Any of the above may be present in varying amounts, some of which are quite small, but nonetheless very effective, weathering agents (eg, ozone concentrations as small as 5×10^{-8} have caused serious defects in a relatively short time.) To complicate matters, they are not only functions of locality, time of day and year, altitude, etc, but they may act in several combinations to produce effects often quite different from those of individual action. For example, the individual effects of gases and moisture is quite different from their combined effect. It might be added here that in most cases of this "combined effect," moisture is always one of the agents involved (*55*).

EFFECTS——Usually the weathering effect on a material produces changes in the surface appearance, but the change may be an invisible one, such as loss of tensile strength, hardness, resilience, etc. These changes may be broadly grouped as "Optical," "Contour," "Physical," and "Miscellaneous," and will include, among many, such terms as light-fading, discoloration, delustering, haze, crazing, cracking, pitting, chalking, blooming sponginess and mildew. (Interesting data on the weathering effect on coatings will be found in publications such as ASTM STP 133.)

A) *Optical.*

1) *Light fading* is perhaps the most common of all weather-

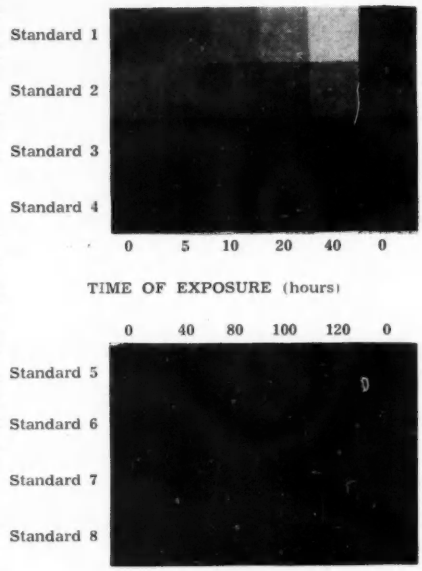

Figure 2
Example of light fading

ing effects on rubber, textiles, and plastics and is characterized by a discoloration at least on the surface. Figure 2 shows degrees of light fading when ISO textile standards have been exposed under carefully controlled conditions. Fading is caused largely by natural sunlight in combination with various amounts of moisture and heat, although similar effects can be caused by gases (*56,64*) with and without moisture.

2) *Delustering* is usually recognized as a loss of original surface luster or gloss and is perhaps caused either by chemical reactions within the material or by external physical or chemical agencies surrounding it or both. In some cases a microscopic pitting effect, different from the originally smoother surface, scatters incident light to reduce gloss.

3) *Crazing*, sometimes called "alligatoring", is caused by the formation of an oxidized film, in the case of rubber, with subsequent leaching and contraction.

4) *Discoloration* is here broadly defined as a distinct change in color or hue, differing from the grayness or saturation (*43*) of a given color, which is largely associated with light fading. It may range from the characteristic yellowing of plastics, such as polyesters, to the blooming or frosting produced when internal solids and volatiles diffuse to the surface of a material.

B) *Contour.*

1) *Cracking*, sometimes called "checking", is the distinct rupture of the surface principally in depth and length and may be in a regular or irregular pattern. The regularity is usually determined by internal or induced stresses: for example, ozone cracking of rubber is always normal to the stress.

2) *Pitting* is characterized by a circular indentation of varying size on the surface. The pits are gouged out of the material by the momentum of incident article, a process very similar to sandblasting.

3) *Porosity* or *sponginess* usually ranges from minute voids to relatively larger ones on the surface.

C) *Physical.* These generally include changes in physical properties, mainly tensile strength, hardness, flexibility, resilience, etc.

D) *Miscellaneous.*

1) *Chalking* usually refers to the release at the surface of particles of mineral filler, etc, and is usually removable by wiping.

2) *Mildew* is beyond the scope of this paper, but it is mentioned to stress its growing importance.

ARTIFICIAL WEATHERING METHODS

Let us now consider possible artificial weathering methods in terms of method concepts, with limitations of apparatus, instrumentation (including some detecting, measuring, and recording) and data evaluation; and compare these possible methods with present methods. This consideration is not essentially outlined by specific tests, in order to avoid duplication, but they are discussed in necessary detail. No "internal" parameters such as a material's particle size, composition, etc, are included in this paper.

GENERAL CONCEPTS OF METHODS, LIMITATIONS——Since natural weather depends upon the time of the day and year; and varies

within and among areas, it is possible to establish what might be called a "standard weather" only for some area. The use of such a "standard weather" is essential in artificial weathering methods and various forms have been used for many years, some as national standards. With respect to solar radiation in an area known for "good weather" throughout the year, namely, the Phoenix, Arizona area, measurements have shown (*34*) that the annual total radiation of the daily average since 1954 has varied only pom five percent approximately. It should be noted, however, that within any of these years, and in a small nontypical number of cases, the radiation varied (*22*) from two to 100 hourly and 100 to 600 daily in langley units (See Appendix I — Definitions). Other areas of the USA are less favorable. There usually exists some pattern and, within this pattern enough fairly constant parameters to justify at least an attempt to formulate the "standard weather" for that area. In England, for example, an "effective humidity" (*52*) of 20 per cent has become the standard for British-used products. In a sunnier country, such as India, the effective humidity would be lower, according to McLaren's work (*52*). However, products suitable for one area may be unsuitable for another within a country. The problem is more complicated for products which may be used anywhere in the world.

By establishing maximum values for the main weathering parameters, a more reliable simulation, or artificial weathering, is assured. But in some cases the maximum value is limited. For example, an abnormally large increase in the intensity of the light source of an artificial weathering tester would most certainly raise the specimen temperature much above that of thermal degradation.

It is important to obtain the best possible duplication of these main weathering parameters, usually sunlight, temperature, and moisture (which includes humidity and rain) as functions of time. Ideally, these time functions should correspond at least approximately to those of the locality, eg, those of summer and winter or of day and night. In short, a cyclic variation rather than a constant value of the parameters is desirable.

A) *Light.* While a "constant" output of light source can be, and in fact is, used to approximate the average of the year or some month, (eg, July noon intensity is often used) an intensity output which is a preselected function of time should be considered, since recent studies indicate that weathering effects of such variable cyclic light intensities differ from those of constant intensity (*17*). Since the variation is sinuoidal, simulation by an artificial light source should not be difficult. No light sources with variable output are now commercially available, but the author's experiments indicate they are possible to produce without undue sacrifice of life and other performance characteristics. Present methods of intensity variation at the sample are not periodic, but are sudden "on-off" exposures, either by turning off the source or turning the specimen. A periodic exposure should, and can, be devised. It is well to remember that ideally accelerated cyclic light exposures should also include the material's chemical and physical reaction characteristics. A given example concerns threshold values and time functions (response, lag, etc) in, say, a photochemical stimulus, ie, from "ground to excited state." More specifically, the reaction may be initiated only after a certain minimum energy is received. And once stimulated, the transient time for increase and decrease to and from the steady state value of the reaction may be considerably more than the accelerated on-or-off period.

The magnitude of the artificial light intensity to be used depends largely on 1) the intensity of natural sunlight to be simulated, and 2) the required acceleration of artificial weathering. For a given source, the intensity at the sample can be varied by the source-to-sample distance. With respect to intensity, suitable light sources are presently available, but their spectral composition does not correspond to that of natural sunlight, except for the xenon lamp with suitable filters.

It is obvious that a good approximation of a spectral composition of natural sunlight is essential (*28,50*), since many materials are critically sensitive only to various portions of the solar spectrum. Notable among these are plastics, which are photchemically responsive to certain bands of the UV region (*37,57*).

Concerning the spectral composition, Figure 1 clearly shows that the carbon-arc-light source, until recently the commonly accepted "standard" in the USA, produces a very poor approximation of natural sunlight with respect to intensity and wavelength, especially in the UV region.

In general, the control of a good artificial light at the sample depends on the source's electrical supply, the characteristics of its filters, and the aging properties. Automatic voltage regulation is essential, yet none is offered in commerically available apparatus. Unsatisfactory means, such as tapped transformers and manual monitoring, should be avoided.

Measurement of the artificial light source's intensity over the spectrum at the sample is essential. If the light detector, such as a photocell or ferrioxalate actinometer (*33*), has a frequency characteristic, the measurement is not too reliable. Pyrheliometers have been used successfully many years by the United States Weather Bureau with good reliability to measure solar radiation in langley units (*21*), but unfortunately they do not account for the effects of other parameters, notably moisture (*56*). A recording potentiometer gives a permanent record. The glass enclosures of the pyrheliometer's thermopile sensor is not frequency-sensitive to solar radiation between 400 to 2400 $m\mu$. (The pyrheliometer measures the temperature difference between its black and white panels; this difference is very nearly proportional to the total radiation in the earth's solar spectrum.)

B) *Temperature.* This natural weather parameter, together with humidity, is very erratic daily, as shown earlier, for a given area and season because it varies with altitude. For these reasons, it is suggested that a constant temperature be used, or, if the above-indicated variable light source is used, the temperature variations from such a source be tolerated about some mean test temperature. It is to be noted that, in general, most chemical reactivity in the weathered sample is an exponential function of the temperature, which is perhaps a good reason to maintain temperatures as indicated above.

Extreme temperatures outside the 40 to 90° Fahrenheit range must be considered, including not only those materials constantly exposed to the weather of the temperate zone, but in more unusual cases: the Arctic and tropics.

Heat sources and temperature controllers are familiar and readily available items, and therefore will not be detailed in this paper. In some testers, the problem is to remove heat usually generated by the light source.

C) *Humidity.* Haynes (*34*) says: "Control and corrective measures are complicated by variations in relative humidity, both diurnally and regionally. It is usually low during the warmest part of the day and high when the temperature has dropped

to its minimum. At high temperatures, a small temperature decrease below the dew point will condense relatively large amounts of water vapor, while at low temperatures, the same decrease below the dew point will produce only slight condensation. In the warmer regions, particularly where the air is moist (along the seacoasts), condensation is greater than in the cooler, drier regions."

As indicated above, daily humidity is very erratic. McLaren (*52*) says: "In textile lightfastness testing, the most important cause of anomalous results is the effect of humidity during exposure."

A constant humidity of suitable value should therefore be used. What this "suitable value" should be will no doubt be argued for all time because it depends not only on the temperature but also on the materials being tested (occasionally some are highly moisture-sensitive). The United Kingdom has established twenty percent effective humidity for Great Britain only after analysis of the data from 187 of its weather stations showed a rather uniform curve of Daily Hours of Bright Sunshine varying inversely with the Effective Humidity (*52*).

Effective humidity is used by United Kingdom because its sunshine is lower and therefore its atmosphere relative humidity is higher than for sunnier countries (eg, India). "Since the moisture content of a textile or other sample is determined by the relative humidity of the air in contact with its surface and, since sunlight makes the sample warmer than the atmosphere, the sample's moisture content (and therefore its fading or weathering) is much more affected by the proportion of sunlight occuring during the exposure period than by the relative humidity of the atmosphere" (*54*). Compare this with what every housewife believes, namely, that a damp or wet fabric hung up to dry in the sun will fade faster than a drier one.

The range of humidity should be controllable at preset amounts from 10 to 100 percent.

Concerning moisture-sensitive materials, it is sometimes advisable to test at two humidities, one dry at 40 percent and the other humid at 85 percent (*74*).

Measurement of atmospheric humidity is generally made by a psychrometer or hygrometer, the advantages and disadvantages of each being about equal. Control by the hygrometer, commonly a hair, plastic, or dewpoint type, is perhaps easier for presently available instrumentation where an electrical signal actuates spray systems to produce humidity at the preset amount.

D) *Rain.* Since the main effect of natural rain is essentially to remove particles from without or within the sample, and since the natural rain pattern is perhaps the most irregular of all, a rain duration per day for various durations would seem advisable. Clean water, preferably distilled, can easily be admitted at a desired flow-rate by a timing system.

E) *Radiation*—See page 34, Miscellaneous: Nuclear Radiation)

EXPOSURE TOPICS———

1) An acceleration method is desirable to save time and money. Today, accelerations of at least eight days to one are used with repetitive results and the only present question is: Is there an upper limit to the acceleration ratio? Data to date are inconclusive but a broad general answer is that such an upper limit does exist under present test conditions and with presently available apparatus.

An estimate of the artificial weathering time, at least with respect to sunlight, can be made from Equation I.

$$L_a = (L_n \text{ DH } I_n) / I_a \qquad (Eq\ I)$$

where

L_a = accelerated service life, hrs
L_n = natural life yrs estimated
D = number of days sunshine per year
H = hours of average sun per day
I_n = average daily sun intensity, foot candles
I_a = artificial source intensity, foot candles

The Prot method should be mentioned here because it has been successfully used to predict service life by accelerated means.

2) *Controls and Standards.* The use of standard samples or controls is recommended for normal test conditions. In abnormal conditions, eg, excessively sensitive materials, more than one type of control may be used; for example, Toepffer (*74*) tests at two different humidities for the same control sample. One great advantage of such controls is that they respond equally with the test specimens to all variations from normal test procedure, ie, to variations in, say, light intensity.

3) *Total Exposure vs Total Exposure Time.* Total exposure of test samples to light irradiation is much better than total exposure time, because of the many variations possible. Such variations can be caused by the light sources' supply voltage variation, its aging characteristics, its filter properties and many others. To illustrate the term "many others," for the filter alone: a) the material composition changes with time because of light, moisture, and other quantities surrounding it; b) solutions touching it will deposit precipitates; c) dirt from the water supply may effect the light transmission, and so on. Perhaps this slight detail (and there are many more for each of the quantities involved) will illustrate the importance of the total exposure concept. The common assumption that irradiation is proportional to time of exposure is definitely wrong and it is such serious assumptions as this which have perhaps produced many of the "anomalous results" reported in even this year's pertinent literature.

4) *Service Life.* For service life, or fitness for purpose, the rate of weathering is usually more important to the weathering technologist than the amount of weathering to which a product will be subjected during its lifetime. During the same estimated service life, two different products, "A" with twice the weathering resistance of "B", will show different weathering rates, so minimum weathering resistance requirements or values should also be determined by service use.

As indicated by Equation I, the accelerated testing time (when a time basis is used) depends on the estimated service life. Incorrect estimates for new materials whose weathering life is unknown can be corrected by trial-and-error application of Equation I until a minimum lightfastness or weathering standard is thereby determined. Although some American industries use this method and therefrom establish minimum weathering standards for various classes of goods, its merit and extended use should be seriously considered for future application (*54*).

In conclusion, some of the above proposals may seem somewhat impractical, perhaps because they have never been implemented, or visionary, perhaps because no one has thought of them before, but it is noteworthy that many of the presumably practical ones are today not used; eg, consider the obvious requirement of a constant voltage to the light source or the concept of total exposure rather than total exposure time.

At the risk of double jeopardy (national and international), I would outline briefly the specifications of the ideal test as this paper has tried to indicate; but enough information has been presented for those courageous ones who are presently dissatisfied with present results.

EVALUATION TOPICS——For general evaluation of all or most of the above weathering effects, the eye seems superior to available instruments, especially when it is supported by good sets of standards, photographs, and similar aids. In some cases, eyes vary, usually more than instruments, but their relative costs usually relegate the instruments to very special cases. In this connection, however, it must be mentioned that errors have occurred, even by experienced visual evaluators, and these errors have often caused lengthy and costly problems both in industry and research.

Eye evaluations of complex weathering effects, eg, fading of textured materials, is better than instrumental evaluations, eg, by reflectance measurements. On the other hand, measurement of gloss by instruments is better than by eye.

Where optical assessments, either by eye or instrument, are impossible or difficult, evaluation may be made by measuring the change in some physical property. For example, light oxidation and embrittlement or cracking of an inorganic material such as rubber can be detected by determination changes in strain under constant load.

BRIEF COMMENTS ON PRESENT ARTIFICIAL WEATHERING METHODS——

A) *Parameters.* The most common type of light source used until recently in the USA was the carbon-arc lamp which, in view of Figure 1, is wholly inadequate for general tests of weathering properties. This is largely because of its poor sunlight approximation, especially in the UV region. But the xenon source with excellent sunlight approximation, both as to intensity and spectrum, is now available. Commenting on it, an eminent light-fading authority says: "Now complete reliability becomes a possibility because results identical with those obtained from a daylight exposure should theoretically be obtained, provided the temperature and moisture content of the specimen are correct."

A comment by McLaren (*53*) in 1957 also supports the xenon lamp: "Now that the xenon lamp has an emission spectrum identical with sunlight for all practical purposes, and whose constant output could be varied to simulate intensities in various parts of the world, the solution to many problems associated with natural sunlight tests is here."

Only xenon lamps of constant output are now commercially available,

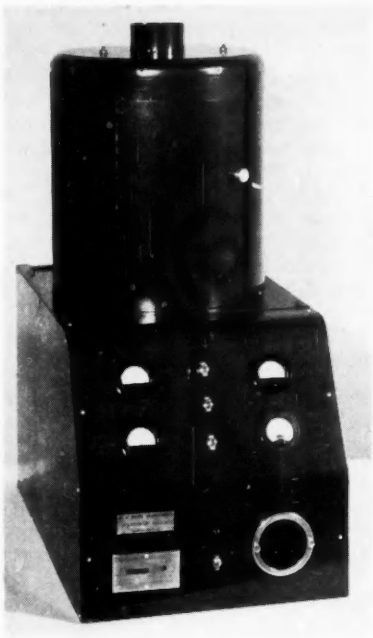

Figure 3
Improved weathering tester Model SSI

but those of variable output may soon be commercially available at which time the author's proposed cyclic exposure could be tried out.

Parameters of constant temperature and humidity for two main classes of materials should be controllable at preset amounts with ideal tolerances of pom one percent of maximum value. Cyclic variation is not recommended. Present temperature in most tests the world over is determined partly by the thermal resistance of the material, eg, plastics should not be subjected to more than 120° Fahrenheit. In general, relatively little consideration is given to use of humid atmospheres. Presumed values at the specimen are usually much higher than actual values, largely because of sample heat (*52*). Actual relative humidity values of approximately 30 percent for normal materials, and 85 percent for moisture-sensitive materials should be used.

EXPOSURE TOPICS——In all exposure methods, the total exposure and not the total exposure time should determine the total light irradiation of the sample, since a given light source usually varies with time. Furthermore, all new light sources vary in original intensity so the present complex calibrations of each of these sources is eliminated if the total exposure concept is used.

Methods of measuring total exposure which involve standard control specimens except paper are satisfactory. (Moistening of standard papers introduces errors.) While the use of a standard plastic avoids this difficulty, instrumentation for measuring spectrophotometrically the reduced transmission through the plastic standard is somewhat awkward, requiring, for example, a constant voltage supply to the light source. An electronic device I call the FCH meter (foot candle hour meter) shown partly in Figure 3 shows as a number the product of the instantaneous intensity and the time, ie, the area under the intensity vs time curve. Variations of intensities due to variations in light source supply voltage, light source aging, absorption of any filters, in fact, any variations are negligible. Exposure measurements on a time basis for such variations introduce grave errors.

EVALUATION——In general, present evaluation methods of weathering effects are largely by visual judgment rather than by instrument evaluation. This is especially true for the more complex effects involving, for example, texture, haze, color, etc. But progress in instrumentation is forward, as shown for example by recent additions in the plastic field. Here measurements by absorption or scattering of light in relatively inexpensive instruments have provided more accurate and absolute results than by visual judgement.

For textiles, the ISO gray scale has proved substantially reliable. It consists of a geometric series of standard values favoring the smaller, earlier changes in lightfastness. Each of four successive grades has a value twice that of the preceding value.

Other new and promising methods of evaluation include those of Hirt (*37,38*), Mudd (*59*), McCarthy (*57*), JSDC (*10*), Baxter et al (*12*), and Butterworth et al (*19*).

APPARATUS——Artificial weathering apparatus should, of course, afford the simplest and most economical realization of at least the most important of the ideal test parameter requirements. Not to be neglected is reliability, reasonable life, and ease of operations, installation, and repair.

Some commercially available weathering apparatus is not satisfactory for the following main reasons:

1) Inadequate approximate of natural sunlight by artificial means.
2) Lack of good general device or method for detecting and indicating total exposure parameters

APPENDIX I — DEFINITIONS

Fastness	— The ability to resist physical and chemical changes which produce an optical weathering effect.
Footcandle	— light illumination unit, 1 fc = 1 lumen/m^2 = 10.76 lux
Lumen	— luminous flux unit = 0.0015 watts
Lux	— light illumination unit = 0.093 fc
Langley	— solar radiation unit = 1 gr cal/cm^2 = 3.69 btu/ft^2
Humidity, effective	— An area humidity related to its average sunshine
Millimicron	— 10^{-3} microns = 10 Angstroms
pom	— plus or minus
Radiation, Solar	— Ultraviolet: solar radiation at earth's surface 200 to 380 mμ Visible: solar radiation at earth's surface 380 to 780 mμ Infrared: solar radiation at earth's surface 780 mμ to ——
Weathering, Natural	— The interaction of natural earth-atmosphere components and a material
Weathering, Artificial	— The interaction of artificial earth atmosphere components and a material under standard, controlled conditions.

of at least sunlight and moisture.

3) Excessive operational variations in parameters; more and better controls needed.
4) Lack of suitable humidity parameter in most units.
5) Lack of gas parameter in all units.

An earlier model of one commercially available tester which includes or satisfies all of the above requirements is shown in Figure 3. It has produced the best correlative data known to date.

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Abstracts

The Versatility of Orlon

Faris, B F, *Dyer 122, 593, 595, Oct 10, 1959*

In dyeing Orlon acrylic fiber there are a number of attributes that are responsible for the fiber's universal acceptance. These properties are brilliance of shade, fastness of dyeings, cross-dyeing opportunities, shade reproducibility, ease of printing, and response to chemical and mechanical finishing.

Properly dyed Orlon shows no fading or staining in 160°F wash tests of any kind, and gives no problems in perspiration or sublimation tests. A lightfastness range of 40-80 Fade-Ometer hours is readily obtained in practically any shade. A wide range of shades can be dyed on Orlon which will withstand 500 hours' exposure in a temperate climate. For this reason it is expected that Orlon dyed with these light-fast colors will find rapid acceptance in outdoor uses, according to the author.

Cationic or basic dyes exhaust completely on Orlon irrespective of the bath-to-goods ratio. A high dyeing temperature (205-212°F) is recommended.

Fabrics of 100% Orlon can be printed with cationic dyes or disperse dyes by batch processes. For many uses, resin-bonded pigments are employed, and are generally the only satisfactory class for printing many blends containing Orlon.

The fiber responds particularly well to mechanical finishing, and is being used in many attractive raised face fabrics. In addition, a number of chemical finishes have been developed, including durable water repellents and antistats.

Some of the more recent developments include suitings of Orlon blended with Dacron polyester fiber and with wool, jersey fabric of Orlon and wool, blends of Orlon and rayon for dresswear (usually yarn-dyed), socks of high-bulk Orlon, blends of Orlon and rayon for heather effects, and hand knitting yarns. Blends of Orlon and Dacron are dyed with a combination of disperse dyes (for the Dacron) and cationic dyes (for the Orlon).

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Processing Cotton Blended with Terylene

Anon, *Man-made Textiles 36, 62, 64, Dec, 1959*

Cotton is blended with Terylene to reduce the electrostatic charges developed by the fabrics, to impart some degree of absorbency, and to produce a more conventional handle than that of an all-Terylene fabric. For these purposes 33% of cotton is sufficient. The inclusion of cellulose does reduce the strength, but 67% of Terylene is sufficient to ensure that the fabrics have higher tear strength than all-cotton ones, increased abrasion resistance, increased recovery from creasing, and lower shrinkage.

Terylene/cotton yarns are commonly sized with a mixture of polyvinyl alcohol with a water-soluble starch; most fabrics, therefore, do not require desizing. Some warps may be sized with PVA/sago mixtures, and these will require a conventional desizing treatment.

Fabrics containing Terylene must not be kier-boiled. They can be scoured in two g/l caustic soda with a suitable detergent, but the temperature must not exceed 60°C. Excessive conditions of scouring with caustic soda solutions should be avoided because damage to the fiber might occur. When soda ash is used the temperature can be raised to 70-80°C.

Mercerizing the fabric improves the dye receptivity of the cotton, minimizes the shrinkage and reduces the soiling of the cotton.

For good dimensional stability to washing, boiling and ironing, fabrics should be set for 30-40 seconds at 180°C. Heat-setting, which should precede dyeing, also reduces the tendency to pill and improves crease recovery. Pilling will still occur if all the surface hair is not removed by cropping or singeing.

Bleaching is required mainly for the cotton component, and any of the conventional bleaching treatments may be safely employed. The very best white is produced by bleaching with chlorite followed by a peroxide treatment. An optical bleach substantive to Terylene (eg, Uvitex ER) improves the white. It may be combined with the chlorite treatment.

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Application of Nuclear Radiation and Radioisotopes to Textile Material and Processes

Rutherford, H A, *Textile Forum 17, 6-7, 13, 16-17, Dec, 1959*

On Nov 1, 1958, the Textile Research Center at the School of Textiles of North Carolina State College undertook a broad study to explore possible applications of nuclear energy and technology to textile materials and processes. The program is being jointly sponsored by the Isotopes Applications Division of the U S Atomic Energy Commission and four textile firms: Burlington Industries, Inc, Cone Mills Corp, Reeves Bros, and J P Stevens & Co.

The research is being conducted along four general lines as follows: 1) Modification of fibers by "naked" exposure to radiation; 2) Modification of fibers by *in situ* polymerization and/or graft polymerization of vinyl monomers; 3) Application of beta-gauges to textile processes; 4) Use of tracers and activation-analysis techniques.

The major emphasis during the first year was placed on the development of techniques for fiber modification by a single monomer, and on the application of beta-gauges for the measurement and control of textile processes.

All fibers are ultimately degraded by exposure to radiation. However, the rate of change in properties differs from fiber to fiber. Some materials, at intermediate points of exposure and prior to showing signs of degradation, undergo changes that may be desirable (eg, cross-linking), which cannot in most cases be carried out by ordinary chemical means.

The exposures of the various materials have been conducted in a Cobalt-60 gamma source of approximately 2300 curies, having a dose rate of 2×10^6 roentgens per hour.

In preliminary work, the rate at which a number of textile materials were changed by gamma radiation was determined. It was also necessary in preliminary work to investigate the behavior of a number of monomers during exposure in the gamma facility.

For several reasons, acrylonitrile was selected as the monomer with which to establish the principles or techniques for the application of vinyl compounds to fibers. Acrylonitrile polymerized most readily of all the substances tried, and its presence on most fibers could be detected very readily by a simple analysis for nitrogen.

Various fibers were exposed to gamma radiation in acrylonitrile vapors saturated with water. It was interesting to note that cotton, mercerized cotton, rayon, acetate, Saran, nylon, and polypropylene, which are

(*continued on page 63*)

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Northern New England Section——

DYEING DACRON BY EMULSIONS OF SOLVENT SOLUTIONS OF DYES*

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A solvent-dyeing system in which a solvent solution of a dye is emulsified in water was found to be a very effective method of dyeing Dacron 54. The effects of dyeing time and temperature, volume of solvent, and concentration of emulsifier were investigated. Several solvents and dyes were considered.

The amounts of disperse dyes combined with Dacron from emulsified toluene solutions of these dyes after one hour of boiling at atmospheric pressure was found to be considerably greater than that obtained from a normal water dyeing at 250°F under pressure.

Pilot-plant dyeings of Dacron top by the solvent system were perfectly level and considerably greater in depth of color than equivalent water dyeings at 250°F.

INTRODUCTION

DYEING of synthetic hydrophobic fibers has been rather difficult ever since their advent. These difficulties can be attributed partly to the lack of attraction forces between the fiber and dye molecules due to the rather inert chemical structure of the polymer molecules in the fibers. In addition, the rates of dyeing and fiber diffusion are low in a water system because the fibers are hydrophobic and do not swell in water.

Numerous methods of increasing the dyeability of synthetic fibers have been introduced. Polymers of some fibers have been modified to obtain fibers containing attraction groups for dye molecules. High temperatures, such as 250 to 300°F, in pressure equipment, have been used in order to increase the rate of fiber diffusion. Carriers have been employed to swell the fiber or decrease the intermolecular attraction forces of the fiber molecules so that the rate of dyeing will be greater. Non-aqueous solvents have been suggested as a dyeing medium for the acrylic and polyester fibers since water is not an ideal solvent in the case of these hydrophobic fibers.

In a study of carrier dyeing, Schuler (*1*) found that the rate of disperse dye penetration in Dacron varied directly, and approximately proportionally, with the amount of the carrier compound adsorbed within the fiber. The only function of the carrier compound in the external dyebath is to control the amount of carrier adsorbed by the fiber. From this he postulated that the weakening of the intermolecular attraction forces of the fiber polymer molecules, created by the presence of the adsorbed carrier molecules, resulted in an increased rate of dye adsorption. Glenz and his coworkers (*2*) obtained results which confirmed the findings of Schuler.

In a study of dyeing in non-aqueous media conducted by Peirent and Sanghrajka (*3*) at Lowell Technological Institute in 1956, it was found that the amount of dye adsorption in a solvent medium was, in some cases, significantly greater than that in a water system of the same dye.

Non-aqueous systems are not generally suitable for practical application. However, alcohol dyeing of acetate, with acid dyes, has been used successfully in industry. The high cost of solvents has, in general, prohibited consideration of the use of solvent dyeing.

As a result of Peirent and Sanghrajka's study of non-aqueous dyeing media, an exploratory study of emulsion dyeing was carried out by Skinkle and Kim (*4*) at LTI in 1958. Their results indicated that an emulsion system for the dyeing of synthetic hydrophobic fibers might be possible.

The present paper reports an investigation of some of the major variables in the dyeing of Dacron by means of a water emulsion of a solvent solution of disperse dyes. The emulsion contains the organic solvent solution of the dye in the internal phase, which is stabilized by the presence of a surface-active agent. Water is used as the external phase, acting more or less as a means for transporting a relatively small quantity of solvent solution of the dye through the relatively large mass of fibers to be dyed. This type of system should be classified as a solvent-dyeing method, as dyeing is believed to take place from the solvent. It differs from the 100%-solvent system in that the solvent does not act as both the dyeing medium and the vehicle for the dye solution. It differs from the carrier method in that the carrier acts essentially on the fiber and is not concerned with the dissolving of the dye.

EXPERIMENTAL

The fiber used in all dyeings was Dacron type 54 raw stock, which had been cut into short lengths. Each dyeing in this investigation was prepared separately, using an analytical balance and volumetric equipment for all weight and volume measurements.

Table I presents the formula used for dyeing Dacron with emulsions of toluene solutions of CI Disperse Red 5 (CI #11110). The weights and volumes which were used are shown in

*Presented at Lowell Technological Institute, Lowell, Mass, on May 15, 1959 before a joint meeting of the Northern New England Section and the LTI Student Chapter.

TABLE I

Emulsion dyeing formula

	Weight or volume	*Percent solution*	*Concentration*
Dacron 54	3.00 g	—	—
Dye (CI Disperse Red 5)	0.0600 g	0.02	0.20 g/l
Solvent (toluene)	15.0 ml	5.0	50 ml/l
Surfactant (Triton X-100)	0.1500 g	0.05	0.5 g/l
Water	285.0 ml	—	—

Volume of dyebath = 300 ml (100:1 volume ratio)
Two percent of dye on fiber weight
Temperature: 212 F; Time: one hour

TABLE II

Water and emulsion dyeings at 212°F and 250°F

Test	*% R*	*K/S*	*Relative concentration*	
			Based on H_2O-212°F	*Based on H_2O-250°F*
H_2O—212 F	8.70	4.79	1	—
H_2O—250 F	3.83	12.17	2.54	1
5% Toluene—212 F*	2.83	16.91	3.53	1.39
5% Toluene—250 F	2.42	19.84	4.15	1.63

*Average of two tests

the first column; concentrations as percent solution and as grams or milliliters per liter are shown in the other two columns. Two percent of dye, based on the fiber weight, was used in all experiments. CI Disperse Red 5 and toluene were used in most of the work. As shown in Table I, 300 ml of the emulsion dyebath contained 15 ml of toluene; this is a 5% toluene content based on volume. This amount of toluene was used in most of the experimental work while the variables of emulsion dyeing were studied. Since it is difficult to agitate raw stock uniformly in a beaker dyeing, a large (100:1) bath ratio was employed.

Ethyl lactate, xylene, and cyclohexanone were studied as solvents under the conditions which were specified for toluene in Table I. In addition to CI Disperse Red 5, emulsion dyeings of 2% of the following dyes were investigated:

CI Disperse Blue 3 (CI 61505)

Eastman Polyester Red 2G

Latyl Blue 4R

CI Disperse Violet 5.

In the emulsion dyeings, 0.0600 g of dye was dissolved in the selected volume of solvent. The solvent solution was then placed in the hopper of a Thomas hand emulsifier. Fifteen hundredths of a gram of Triton X-100, dissolved in sufficient water to produce a final dyebath volume of 300 ml, was also added to the hopper. This mixture was then emulsified by pumping it through the emulsifier. The emulsion was placed in a beaker, which was covered with a round-bottomed flask through which cold water was circulated to prevent evaporation of the dyebath. The emulsion bath was brought to the boil, at which time the fibers were entered. Boiling was considered for one hour, during which time the bath was agitated periodically. The dyed samples were removed, washed thoroughly with water, and dried in a Freas forced-draft oven.

To obtain standard water dyeings to which the emulsion dyeings could be compared, each disperse dye selected was applied by a method which is identical to the emulsion dyeing except that the 0.0600 g of dye were pasted with 0.1500 g of a nonionic surfactant, Triton X-100. This paste was gradually diluted with water to a final volume of 300 ml. The fibers were entered at the boil and dyed at the boil for one hour.

High-temperature dyeings were identical to the water and emulsion methods except that, when the Dacron fibers were added to the boiling dyebath, the beaker was placed in a pressure cooker containing water which had been preheated to 212°F. The pressure cooker was sealed, heated to 250°F in 15 minutes, and maintained at this temperature for 45 minutes. The pressure cooker was then cooled; the samples were removed, rinsed, and dried.

The Kebelka-Munk function of

$$\frac{(1\text{-Reflectance})^2}{(2)\ (\text{Reflectance})}$$

was employed to determine dye concentration on the fibers. The value of this function has been shown to be proportional to the concentration of dye on fibers when they are dyed by a single dye. If there is no variation in the physical condition of the fibers, the ratio of Kebelka Munk (or the K/S values) is equal to the ratio of dye concentrations in the fibers. In this work, two ratios are used: 1) that which is based on the water dyeing at the boil under atmospheric pressure, and 2) that based on the water dyeing at 250°F under 15 psi pressure. The first method is more desirable for use, but is usually not suitable for Dacron; the second is a commercially acceptable method, but requires special apparatus.

The reflectance values required for the calculation of the K/S function were obtained by measurement on a General Electric Recording Spectrophotometer. The fiber pad samples were prepared in a standardized manner so as to have a variation in reflectance not greater than 0.3%. The reflectance values contained in this study are the average of three measurements taken at the wavelength of maximum absorption of the dyed samples.

RESULTS AND DISCUSSION

The data and results in Table II show a comparison of water dyeings at 212°F and 250°F, and two toluene emulsion dyeings at the same temperatures. The second column shows that the reflectance of the dyed materials varied from 8.70% for water at 212°F to 2.42% for the emulsion dyeing at 250°F. Thus, it is seen that there is a very marked increase in depth of color from water at 212°F to the toluene emulsion at 250°F. The Kebelka-Munk or K/S values are shown in the second column. Column three shows the relative concentration on each of the dyed samples compared to water at 212°F as a standard equal to one. If the Kebelka-Munk value of 12.17, for the water dyeing at 250°F, is divided by 4.79, which is the value of the water dyeing at 212°F, a relative concentration of 2.54 is obtained for the pressure dyeing. This means that there is 2.54 times as much dye on the pressure-dyed material as is on the 212°F water dyeing. The relative concentration of dye on the emulsion-dyed sample at 212°F equals 3.53, and on the pressure-dyed emulsion, the relative concentration is 4.15. These values show that the emulsion dyeings produce a tremendous increase over the water dyeing at 212°F.

The fourth column shows the relative concentrations of the emulsion dyeings compared with the 250°F water dyeing as a standard equal to one. Here it is seen that the emulsion dyeing at 212°F gives a 39% greater concentration of dye on the fibers because there is a relative concentration value of 1.39. The relative con-

TABLE III

Effects of temperature and time on emulsion dyeings

Time (min)	Temperature 140°F	176°F	212°F	250°F
	DYE ON FIBER RELATIVE TO WATER AT 212°F			
15			1.77	
30			1.78	
60	0.68	1.07	3.53	4.15
120			4.76	
	DYE ON FIBER RELATIVE TO WATER AT 250°F			
15			0.70	
30			0.70	
60	0.27	0.42	1.39	1.63
120			1.87	

TABLE IV

Effects of solvent/water ratio and surfactant concentration

Emulsion (% toluene)	% Surface-active agent 0.0375	0.0500	0.0625
	DYE* ON FIBER RELATIVE TO WATER AT 212°F		
2.5		4.54	
5.0	2.96	3.53	3.67
10.0		1.06	
	DYE* ON FIBER RELATIVE TO WATER AT 250°F		
2.5		1.78	
5.0	1.16	1.39	1.44
10.0		2.42	

*2% CI Disperse Red 5 (owf)

centration of the emulsion dyeing at 250°F was equal to 1.63, a 63% increase over the 250°F water dyeing. The results of the relative concentrations shown in this table clearly indicate that a 5% toluene emulsion dyeing of Dacron could be an advantageous method of dyeing.

The effects of dyeing time and temperature on the amount of dye combining from a 5% toluene emulsion are shown in Table III. The results show the relative dye concentration on the fibers compared to both the water dyeing at 212°F and the 250°F water dyeing.

Increasing the dyeing time from 15 minutes to 120 minutes with the 212° F emulsion increased the relative concentration of dye on the fiber from 1.77 to 4.76, based on the water dyeing at 212°F. Since the values for the intermediate times of 30 and 60 minutes are 1.78 and 3.53, dyeing equilibrium was not reached in one hour of dyeing, and it may not be reached in two hours of dyeing.

Examination of the values relative to the 250°F dyeing shows that over 30 minutes of dyeing with the toluene emulsion system is needed to equal or better the yield of 250°F water dyeing. At 30 minutes, the emulsion concentration was 0.70; at 60 minutes it was 1.39, and at the end of 120 minutes, a relative concentration of 1.87 was obtained.

One-hour emulsion dyeings at 140° F (or 60°C), 176°F (or 80°C), and 212°F (or 100°C) show that an emulsion dyeing at 176°F is equivalent to a water dyeing at 212°F, the relative concentration being 1.07. Also, an emulsion dyeing at 212°F was better than the yield of a water dyeing at 250°F. One hour at 212°F gave a relative concentration of 1.39, indicating the emulsion exhausted 39% more dye on the fiber than did a 250°F water dyeing.

A greater increase resulted from the emulsion dyeing at 250°F where the concentration relative to the water dyeing at 250°F was 1.63, a 63% increase. However, further study would be necessary to establish the merits of an emulsion system under pressure.

Table IV presents the results of the study of variations in the concentration of the surface-active agent used to stabilize the emulsion, and variations in the amount of solvent used in the emulsion. The emulsions of 5% toluene with 0.0375%, 0.0500%, and 0.0625% surface-active agent all proved to be sufficiently stable for use in dyeing. When the concentrations of the dyeings for these three emulsions were calculated, it was found that the dye concentrations on the fibers varied directly with the surface-active agent concentration. Relative to the 212°F water dyeing, the results were 2.96, 3.53, and 3.67; compared with the 250°F standard, the results were 1.16, 1.39, and 1.44.

The differences at these surface-active-agent concentrations are large for the small changes in the concentration of the surfactant. This seems to indicate that the increased stabilization of the emulsion, which could be expected as a result of the increased surface-active-agent content, causes more dye to be exhausted onto the fibers. Although small additional increases in surface-active-agent concentration may give further increases in dye adsorption, it is expected that retarding of dye adsorption would result if much higher surface-active-agent concentrations were employed.

The amount of dye on the fibers is shown to vary inversely with the amount or concentration of toluene in the emulsion. A decrease in the toluene concentration from 10.0% to 5.0% to 2.5% increased the dye concentration from 0.42 to 1.39 to 1.78 times the amount of dye on a pressure-dyed sample at 250°F. This is a very large change, and probably the most important controllable variable of the emulsion process. A 40% increase in dyeing yield resulted when the concentration of toluene was reduced from 5.0% to 2.5%. Since the least amount of solvent gave the greatest yield, it is suggested, but not proven, that the optimum amount of solvent may be that amount which is sufficient to form a saturated solution of dye.

Although the mechanism of dyeing was not investigated directly, the effect of the concentration of solvent supports the premise of the work: that dyeing from solvent emulsions is essentially a solvent dyeing, and not a "carrier" type of dyeing. If the solvent were functioning as a carrier under conditions where equilibria were not reached, then the amount of dye combined would increase as the amount of solvent increased. The effect in the emulsion dyeings was exactly the reverse of this.

The results of dyeing with five disperse dyes from a 5% toluene emulsion at the boil for one hour are shown in Table V. The concentrations shown are relative to water dyeings at 250°F. The concentration of each of the dyes combined with the fiber from the solvent emulsions is considerably greater than that obtained by the pressure dyeings. The amount of the increase over the pressure dyeing by the toluene emulsion varied considerably with the specific dye. Eastman Polyester Red 2G yielded 2.81 times the pressure dyeing, and CI Disperse Violet 5 gave 4.75 times the yield of its pressure dyeing. CI Disperse Blue 3 equalled 1.82, and Latyl Blue 4R equalled 1.34. There are two possible explanations for this large difference in adsorption of the individual dyes: 1) it may be dependent primarily on the dye affinity for the fiber, 2) it may be a function of the dye solubility in toluene. Since it was previously shown that lowering the solvent/water ratio increased dye adsorption, the three dyes of lower yield might give greater concentrations of dye on the fiber if the amount of solvent used were made smaller. If there was any cor-

TABLE V

Effect of dyes and solvents on an emulsion system

Dye	Emulsion: Toluene	Xylene	Cyclohexanone	Ethyl lactate
	DYE ON FIBER RELATIVE TO WATER AT 250°F			
CI Disperse Red 5	3.53	1.57	3.52	1.80
	DYE ON FIBER RELATIVE TO WATER AT 250°F			
CI Disperse Red 5	1.39	0.62	1.39	0.70
Eastman Polyester Red 2G	2.81			
Latyl Blue 4R	1.34			
CI Disperse Blue 3	1.82			
CI Disperse Violet 5	4.75			

TABLE VI

Effect of solvent/water ratio and dye concentration

Emulsion (% toluene)	DYE ON FIBER RELATIVE TO WATER AT 212°F: 1% Dye*	2% Dye*	4% Dye*
2.5		1.79**	
5.0	1.16	1.39	2.07**
10.0		0.42	

*CI Disperse Red 5
**Initial concentration of dye in toluene solution phase of emulsion = 8 g/l

relation between solvent solubility and the amount of dye combining as was previously suggested, then each dye would have a different optimum solvent concentration. Such a series of dyes as is shown in the table would then be expected to show large variations in dye concentration on the fiber masses so long as all were applied with the same amount of solvent present. Further investigation is necessary to establish the validity of this suggestion .

CI Disperse Red 5 was applied from 5% emulsions of xylene, cyclohexanone, and ethyl lactate, using the method of the toluene emulsions. The concentrations of dye resulting from these dyeings, relative to a 250° F dyeing as one, are also shown in Table V. Under the one set of conditions employed, the toluene and cyclohexanone results were equal; the relative concentrations being 1.39 times a 250°F dyeing. Xylene and ethyl lactate proved considerably inferior to the pressure dyeing.

Since both toluene and cyclohexanone work well, it seems likely that other solvents might also prove feasible. Again, it is possible that the specific dyeing properties of the solvents may be related, in part, to the dye solubility.

The results presented in Table VI show that the amount of CI Disperse Red 5 combining with Dacron increased as the total dye was increased from one to two to four percent of dye. This is normal for a dyeing system when below the saturation value of the fiber. However, when the concentration of dye in the solvent phase of the emulsion was eight grams per liter in both the four percent dyeing with five percent toluene and the two percent dyeing with 2.5% toluene, the amounts of dye combining with the fiber were nearly equal, 1.78 and 2.08. Since the total amount of dye in the four percent dyeing was twice that of the two percent, the solvent solution concentration appears to be a major factor affecting the exhaustion of dye. If these dyeings reacted as a normal water system, the two percent dyeing would have yielded approximately half as much dye on the fiber as the four percent dyeing.

Cross sections of fibers from the laboratory dyeings with CI Disperse Red 5 at 212°F, under pressure at 250°F, and by means of the five percent toluene emulsion were examined microscopically. These qualitative examinations showed the emulsion dyeing to be more uniformly and thoroughly penetrated than either of the other two dyeings.

Qualitative experiments in pilot-plant dyeing of Dacron top, using a laboratory top-dyeing machine equipped for high-temperature dyeing under pressure, were also performed. Three and one-half pound tops of Dacron 54 were dyed by the five percent toluene emulsion system with CI Disperse Red 5 and with CI Disperse Violet 5. Dyeings were carried out for one hour at 208°F. Both of these dyeings were perfectly level in color and heavier than corresponding beaker dyeings by the emulsion system.

A 3.5-lb sample of Dacron 54 was also dyed at high temperature in the top machine with the same quantity of CI Disperse Violet 5 using the normal water system. The dyeing was carried out for one hour at 250° F under 15 psi pressure. When this top was compared with the emulsion dyeing, it was found that the emulsion dyeing was much heavier than the pressure dyeing at 250°F.

CONCLUSIONS

A water-emulsion system containing a disperse dye, dissolved in toluene as the internal phase, has been shown to be a very effective method of dyeing Dacron 54. With the five dyes studied in the investigation, the emulsion system produced from 1.3 to four times more dye on the fiber than a pressure dyeing at 250°F in water.

The amount of solvent used was shown to be the most important variable in the process. In this study, the greatest amount of dye which combined with the fiber was obtained when 2.5% toluene was used to dissolve two percent of dye, based on the fiber weight. However, there are indications that the optimum amount of solvent is that which is just adequate to dissolve the dye.

A temperature of 212°F and a time of dyeing equal to one hour are necessary to give results equal to or better than a water-pressure dyeing at 250°F for one hour.

A surface-active agent concentration of 0.0625% solution was a little better than lower concentrations. However, concentrations as low as 0.0375% solution produced sufficiently stable emulsions for dyeing and satisfactory dye yield.

Cyclohexanone as a solvent worked as well as toluene with one dye studied, and appears to be a second solvent suitable for emulsion dyeing. Xylene and ethyl lactate produced results inferior to pressure dyeings, but these solvents were better than a straight water dyeing at the boil.

Indirect evidence indicates the process to be a true solvent dyeing process in which dye transfers from the internal solvent phase of the emulsion to the fiber.

It is believed that the principle of emulsion dyeing, which employs water for transporting a relatively small quantity of a solvent solution of a dye through the fiber mass, offers a practical approach to the solvent dyeing of fibers by many types of solvent-soluble dyes, and especially to the dyeing of hydrophobic fibers with disperse-type dyes.

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38th National Convention———

DYEING AND FINISHING SPECIAL BLENDS OF ARNEL WITH ACRYLICS AND ARNEL WITH COTTON IN CIRCULAR KNIT APPLICATION*

Part I

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A compatible dye system has been developed for applying selected neutral-dyeing cationic dyes to Orlon and disperse dyes to Arnel simultaneously. An anionic surfactant, control of pH, and dilution of dyes are used in this system. A mechanism is proposed for the anionic surfactant—cationic dye system.

Practical procedures are given for dyeing reserve, cross dye, and union shades on Arnel/Orlon tubular knits. A range of cationic and disperse dyes are recommended for this blend. Finishing of Arnel/Orlon jersey is discussed.

Principles used in dyeing Arnel/Acrilan blends are reviewed.

Arnel/cotton knit fabrics can be dyed a complete range of shades by a simple one-bath procedure using disperse dyes for Arnel and after-treated direct dyes for cotton. Dyes and procedures used on this blend are given. Union blacks are made with developed dyes. For many applications Arnel affords sufficient dimensional stability so that only an anticurl finish is used. Resin finishing can be used to modify the hand and stabilize the cotton.

INTRODUCTION

THE knit fabric industry has experienced a very rapid growth in the past twelve years and the future appears even more promising. The total value of apparel fabric shipments accounted for by knit fabrics rose from 12.8% in 1947 to over 30% in 1957 (*1*). Knit fabrics are being used in applications where woven fabrics were once used almost exclusively. A prime example is the trend in sport shirts from woven to knits.

What has brought about this accelerated growth? There are several factors such as:

1) Increased awareness by the knit-goods producer as to potential markets and applications for knit fabrics.
2) Consumer demand for improved aesthetic properties, ease of care, and styling in apparel wear.
3) New and more versatile knitting equipment as well as advances in knitting technology.
4) Improved dyeing and finishing techniques.
5) New fiber and yarn developments.

These last two factors have played a major role in this growth. As we all know, dyeing and finishing can "make or break" a fabric. The dyer, through his technological advances, can now convert greige goods made from any of the synthetic fiber blends into a colorful array of highly styled fashion fabrics in heathers, solids and irridescent effects. These advances have given the color stylist more freedom and versatility in designing and styling fabrics for the consumer.

Through chemical and mechanical finishing, the finisher has improved the dimensional stability of knit fabrics, decreased pilling, and provided a variety of hands, textures and surface effects. The fiber producer and textile manufacturer have added to the basic tools of the trade by developing new fibers and yarn blends which give improved functional and aesthetic properties to knit fabrics.

Arnel triacetate fiber offers to the knitting trade for the first time an economical blending fiber which is inherently dimensionally stable. In blends with cotton and the acrylics, Arnel contributes to knit fabrics the following properties:

1) Dimensional Stability
2) Shape Retention
3) Pill Resistance
4) Ease of Care Properties
5) Color Styling through Piece Dyeing.

In this paper, we will describe how Arnel in blends offers flexibility in color styling through piece dyeing. The following items will be discussed:

a) The development of a one-bath process for dyeing union shades on Arnel/Orlon blends.
b) Practical dyehouse processing of Arnel/Orlon tubular knit fabrics.
c) Principles used in dyeing Arnel/Acrilan blends.
d) Dyeing and finishing Arnel/cotton tubular knit fabrics.

PROPERTIES OF ARNEL

Before discussing the dyeing and finishing of Arnel blends, it would be well to examine some of the more pertinent physical and chemical properties of Arnel which make it desirable for circular knit applications.

Arnel is inherently pill resistant because of its fiber tenacity, modulus, and cross-sectional shape. These physical properties have been previously reported. Pilling, which occurs on fabrics made from the polyamides, polyesters, and polyacrylics, is objectionable because the pill balls are held on the fabric surface. In the 50/50 Arnel/Orlon high bulk blend, 40% of the fiber is a high-shrink fiber, which migrates to the core of the yarn bundle during dyeing. The Arnel, which does not shrink, migrates to the surface. In dyed Arnel/Orlon knit fabrics, there is a predominance of Arnel on the surface of the fabric. The Arnel allows any pills which may form to break off; therefore, a pill-free fabric surface is maintained.

As a result of the extremely low fiber shrinkage of Arnel, which is less than 1%, improvements in dimensional stability can be obtained in knit fabrics containing 50% or more Arnel. The degree of improvement is dependent upon the percentage of Arnel used and the fabric construction.

The Second Order Transition Temperature of a fiber is that temperature at which initial softening of the fiber begins. Rapid cooling down from temperatures above the Second Order Transition Temperature can

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TABLE I
Dyeing properties of disperse dyes on 50/50 Arnel/Orlon blends
TWO HOURS AT BOIL

Dye	Staining on Orlon: 0.5% Dye (owf)	3.0% Dye (owf)
Yellows:		
CI Disperse Yellow 37	very slight	moderate
Latyl Yellow 3G	light	light
CI Disperse Yellow 3	light	moderate
CI Disperse Yellow 33	slight	moderate
Lenra Yellow R	none	light
Eastman Fast Yellow 2RGLF	none	light
Foron Orange GFL	light	light-moderate
CI Disperse Orange 16	light	moderate
Reds:		
CI Disperse Red 35	light	moderate-heavy
Eastone Red 2BGLF	light	moderate-heavy
Eastone Fast Pink BGLF	light	moderate-heavy
Eastone Fast Pink 3BGLF	slight	moderate-heavy
Genacron Red B	light	moderate
CI Disperse Red 5	light	moderate-heavy
Latyl Scarlet FG	light	moderate-heavy
CI Disperse Red 7	light	moderate
CI Disperse Red 27	slight	light
CI Disperse Red 28	light	moderate
Latyl Cerise B	light	moderate
Blues:		
Latyl Blue BG	slight	slight
Eastman Fast Blue BGLF	slight	light
Eastman Blue GP	light	moderate
Interchem Acetate Blue NBNJ	light	moderate
Interchem Blue NSP	light	moderate
CI Disperse Blue 3	light	moderate-heavy
CI Disperse Blue 7	light	moderate
CI Disperse Blue 1	Dyes almost a union on the blend	

TABLE II
Staining properties of cationic dyes on Arnel in Arnel/Orlon blends
(2% DYE OWF—TWO HOURS AT BOIL—pH 4.0-5.0)

Dye	Arnel staining: Before clearing	After clearing
Sevron Yellow 3RL	slight	none
CI Basic Yellow 11	slight	none
CI Basic Yellow 21	light	none
Sevron Orange G	light	slight
Basacryl Yellow 420	slight	none
CI Basic Red 14	slight	none
Sevron Brilliant Red B	light	none
Sevron Red L	light-moderate	light
CI Basic Blue 21	slight	none
CI Basic Blue 22	slight	none
Sevron Blue NF	slight	none
Deorlene Blue 5G	light	none
CI Basic Violet 14	moderate	slight
CI Basic Green 4	moderate	slight
Basic Black PSR	very heavy	heavy
Basacryl Blue 214	very heavy	heavy

result in setting deformations into the fabric. The wet Second Order Transition Temperature of Arnel is 205°-212°F; therefore, any softening of the fiber which may occur during dyeing is just in its initial stage. The acrylics which have Second Order Transition Temperatures in the range of 140°-180°F (2) are very susceptible to the formation of winch cracks or creases in the dyeing operation unless extra care is observed during cooling down. Even with due caution, some winch cracks or creases do occur. In the 50/50 Arnel/Orlon blend, the Arnel is relatively resistant to thermal distortion and thus reduces the frequency and severity of winch cracks or creases. This has been confirmed during the past season's production of this fabric. To the dyer this means less rehandles.

Arnel is dyed only with disperse dyes and selected azoic combinations. Arnel has little or no affinity for the cationic dyes used for Orlon and direct cotton dyes; thus, in blends with Orlon or with cotton, a wide range of color styling can be achieved through piece dyeing.

DEVELOPMENT OF ONE-BATH PROCESS FOR ARNEL/ORLON BLENDS

The commercial Arnel/Orlon blend is a 50/50 high bulk blend. This blend is being made into both jersey and sweater fabrics for such applications as knit shirts, blouses, dresses, bulky knit sweaters and brushed and sheared sweaters.

Initial Arnel/Orlon fabrics were dyed by a two-bath procedure in which the Orlon was dyed first with cationic dyes by conventional procedures at a low pH and then the Arnel was dyed with disperse dyes in a new bath. Under commercial conditions, dyeing times ranged from 12-15 hours, and thus the dyeing was quite expensive. Also some difficulty in shade control was experienced as the result of shade wash down and staining of the disperse dyes on Orlon.

In an effort to improve dyeing economics, work was directed toward the development of a one-bath procedure for dyeing Arnel/Orlon blends. Reports from within the trade indicated that the use of cationic dyes and disperse dyes in the same bath had met with some success on other fiber blends. Bidgood and Bell (3) recently reported on the dyeing of Dacron/Orlon blends with disperse and cationic dyes in the same bath.

DYE-SELECTION ——— Dye selection was investigated initially. Although Orlon can be dyed with disperse dyes, in Arnel/Orlon blends the specificity of disperse dyes greatly favors the Arnel. Disperse dye selection is necessary for better shade control and color matching. Table I shows the amount of staining obtained on the Orlon when dyed in the presence of Arnel with disperse dyes.

The disperse azo reds have a greater tendency to stain the Orlon than do the anthraquinone blue and diphenylamine yellow dyes. CI Disperse Blue 1 is novel in that it will dye the blend almost to a union. It could serve as a basis for particularly heavy blue shades, such as royal and navy.

Over sixty cationic and older-type basic dyes were screened for their staining properties on Arnel in the 50/50 Arnel/Orlon blend. These dyes were applied according to the manufacturer's recommendations, which usually specified an acid pH of 4.0-5.0 and the use of a nonionic surfactant. Many of the cationic dyes in a complete range of shades gave little or no staining on the Arnel. With these dyes, any stain left on the Arnel was easily removed with a cold acid-chlorine strip. There were a few of the older basic dyes which stained the Arnel quite heavily and resisted clearing; therefore, indiscriminate selection of the cationic or basic dyes could result in dyeings with poor lightfastness because of the fugitive nature of the cationic dye stains on Arnel. Table II shows the staining effect of a few of the typical cationic dyes on Arnel.

For our developmen. work on a one-bath procedure, a pH range of 6.0-7.5 was chosen initially as a standard condition for the following reasons. Disperse anthraquinone dye structures commonly used for Arnel contain acidic and/or basic groupings. At more acid or alkaline conditions these groups form salts which increase the solubility of the dye in the aqueous solution and decrease its affinity for Arnel. At the moderately acid pH's some of the anionic dispersing agents commonly used in disperse dyes lose their dispersing power. Certain gas-fast disperse blues exhibit tendencies to precipitate at acid pH's due to a loss of the dispersing action. Generally speaking, the optimum pH range for applying

TABLE III

Effect of surfactants on dyebath stability

2% SURFACTANT (OWF)—LIQUOR RATIO 40:1—pH 6.8

Dye Recipe

0.75% CI Basic Blue 21
0.58% Sevron Blue 5G
0.40% Sevron Red L
0.06% Sevron Orange G
1.5% Interchem Direct Acetate Navy Blue NVY
0.8% Eastman Blue GP
0.4% Eastone Red 2BGLF

Surfactant	*Bath stability*
Phosphated long-chain alcohol	unstable
Sodium salt of sulfonated naphthol formaldehyde condensate	stable
Polyethylene oxide chain condensate	unstable
Nonionic surfactant A	unstable
Long-chain alcohol sulfate	unstable
Polyethylene ether of fatty acid	unstable
Sulfonated fatty ester	stable
Aliphatic ethylene oxide condensate	unstable
Nonionic surfactant B	unstable
$C_{17}H_{33}$-CO-N(CH_2)-C_2H_4-SO_3Na	stable

TABLE IV

Retarding effect of certain anionic surfactants

Relative shade depth———Visual

(RUN THREE HOURS AT BOIL—UNION NAVY SHADE—30:1)

	% *Retardation*	
% *Surfactant (owf)*	*Cationic dyes*	*Disperse dyes*
Igepon T [$C_{17}H_{33}CON(CH_3)C_2H_4SO_3Na$]		
1	0	0
6	5-10	0
Parapon AP (sulfated fatty ester)		
1	15	10
6	40	15
Tamol N (neutral sodium salt of a complex condensed aryl organic acid)		
1	20	20
6	70	40

disperse dyes to Arnel is pH of 6.0-8.0. The literature (*4,5*) indicates that there are many cationic dyes which are stable and have good dyeing properties at neutral pH's.

SURFACTANTS——Low concentrations of dispersing agents, such as sodium lignosulfonate, and the alkyl aryl sulfonates, which are commonly used in the manufacture of disperse dyes do not cause instability of dyebaths containing typical cationic dyes, eg, CI Basic Red 14 (*6*). Table III shows the results of a preliminary screening of surfactants to determine their effect upon the stability of a dyebath containing a high concentration of disperse and neutral-dyeing cationic dyes. A dye recipe for a navy shade was used. The solutions were boiled in stainless-steel beakers to simulate conditions encountered in a dye winch. No fabric was present.

These severe conditions indicated that there were some anionic surfactants, ie, Igepon T, Tamol N, and Parapon AP, which promoted dyebath stability. Further work with the surfactants showed that Tamol N exhibited a severe retarding effect on both the cationic and disperse dyes as the concentration of surfactant increased. Parapon AP showed a moderate retarding effect toward the cationic dyes. Igepon T had a slight retarding effect on the cationic dyes; however, for all practical purposes the cationic dyes exhausted completely during normal dyeing cycles. Table IV shows the relative retarding effect of the surfactants evaluated after three hours at dyeing temperature.

Figures 1 through 4 show the retarding effect on the color difference of tertiary shades dyed on both fibers in the same bath in the presence of anionic Igepon T versus a nonionic surfactant. The Arnel was dyed grey and the Orlon was dyed brown. Samples were taken at various time intervals throughout the dyeing cycle. Readings were taken on the dyed samples using the Gardner Automatic Color Difference Meter. The total color difference, $\triangle$ E, between the scoured only and dyed samples was calculated using the formula for total color difference;

$$\triangle E = \sqrt{\triangle L^2 + \triangle a^2 + \triangle b^2}$$

$\triangle$ E was plotted against the dyeing time. The distance between the two curves for any one fiber is an absolute expression of the relative color difference between the sample dyed with Igepon versus the sample dyed with a nonionic at any time during the dyeing cycle.

In Figure 1, where 0.5% surfactant (owf) is used, the dyeing on the Orlon made with Igepon T shows a slight initial retarding effect; however, after 180 minutes at a boil, there is little difference in the shade of the Orlon dyed with Igepon T compared with the Orlon dyed with the nonionic.

At the 5.0% level of surfactant on the Orlon, Figure 2, the dyeing made with Igepon T shows slight retardation after three hours at a boil. Visually the cationic dyes have been retarded about 10% with the high concentration of Igepon T.

Figure 3 shows that even a 0.5% of a nonionc surfactant exhibits a slight-to-moderate retarding effect on the disperse dye for Arnel after three hours at a boil. With 5.0% of nonionic surfactant (Figure 4), retardation of the disperse dyes is very noticeable. The difference shown in the two curves in Figure 4 represents a visual difference of about 20-25% in shade depth. The retardation of disperse dyes, particularly certain anthraquinone blues, is due to their increased solubility in nonionic surfactant agents which causes a shift in the partition coefficient (*7*). The data presented in these curves correspond to what would be expected from a visual observation of the dyed samples.

When 1.0% (owf) of Igepon T is placed into cold dyebaths containing 2.0% (owf) of the various neutral-dyeing cationic dyes, ie, Sevron Orange G, Basacryl Yellow 420, CI Basic Red 14, Sevron Red B, CI Basic Blue 21, CI Basic Blue 22, and Sevron Blue 5G, clouding of the solution is noted; however, no precipitation occurs. When these solutions are heated, they become clear except for Sevron Orange G and Basacryl Yellow 420, which remain slightly cloudy. Two commonly used nonionic surfactants were tested for their effect upon solutions of the above dyes. The cold dyebaths with these nonionic surfactants were clear. As the dyebaths were heated, one of the nonionics produced a cloudy condition in all the baths, probably due to its reverse solubility. The other nonionic did not show this reverse solubility effect. The dyebaths containing Sevron Orange G produced a cloudy condition with both of the nonionic surfactants as well as with the Igepon T, which would indicate that the clouding is due more to the inherent solubility properties of this dye than to the effect of the surfactant present.

DYEING MECHANISM——These data strongly suggest that the following mechanism occurs when dyeing with cationic dyes in the presence of a selected anionic surfactant. In Figure 5 it can be seen that the dye cation forms a loosely held association compound with the surfactant anion. This compound is in equilibrium with the dye cations and surfactant anions.

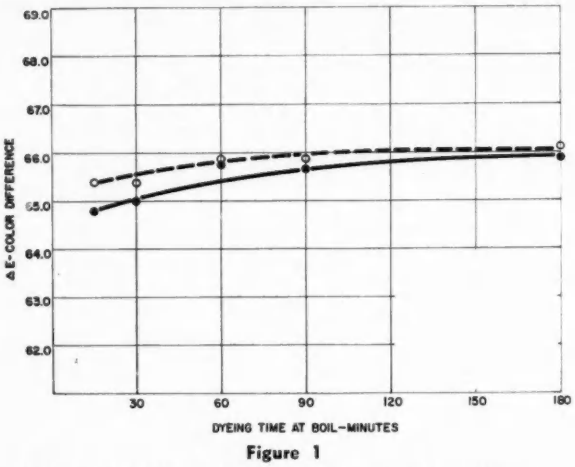

Figure 1

Effect of 0.5% (owf) of surfactants on color difference of Orlon. (Solid line represents Igepon T; broken line indicates nonionic).

Dyed with:
0.8% Eastman Fast Blue BGLF
0.2% CI Disperse Red 35
0.2% Interchem Acetate Yellow HDLF-40
0.5% CI Basic Blue 22
0.2% Sevron Red 4G
0.2% Sevron Orange G

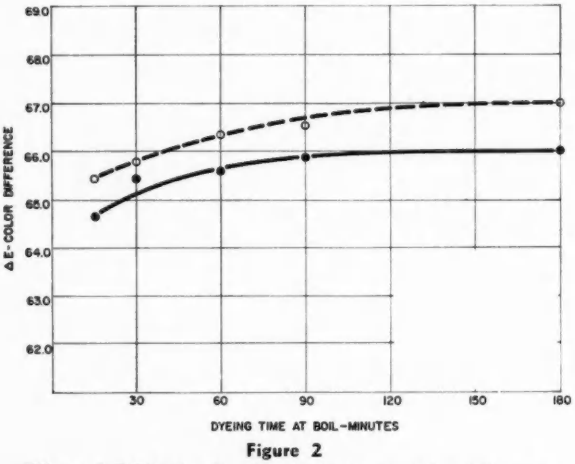

Figure 2

Effect of 5.0% (owf) of surfactants on color difference of Orlon. (Solid line represents Igepon T; broken line indicates nonionic).

Dyed with:
0.8% Eastman Fast Blue BGLF
0.2% CI Disperse Red 35
0.2% Interchem Acetate Yellow HDLF-40
0.5% CI Basic Blue 22
0.2% Sevron Red 4G
0.2% Sevron Orange G

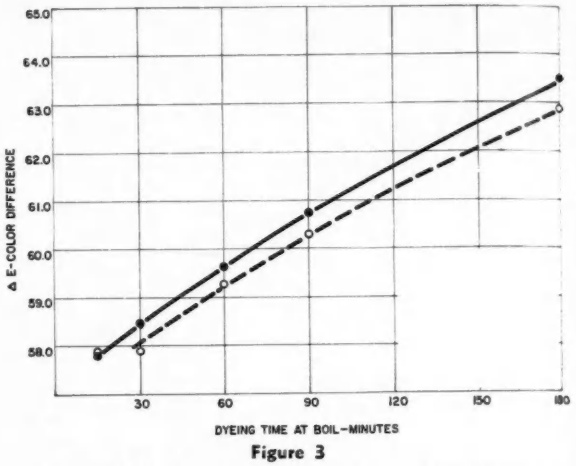

Figure 3

Effect of 0.5% (owf) of surfactants on color difference of Arnel. (Solid line represents Igepon T; broken line indicates nonionic).

Dyed with:
0.8% Eastman Fast Blue BGLF
0.2% CI Disperse Red 35
0.2% Interchem Acetate Yellow HDLF-40
0.5% CI Basic Blue 22
0.2% Sevron Red 4G
0.2% Sevron Orange G

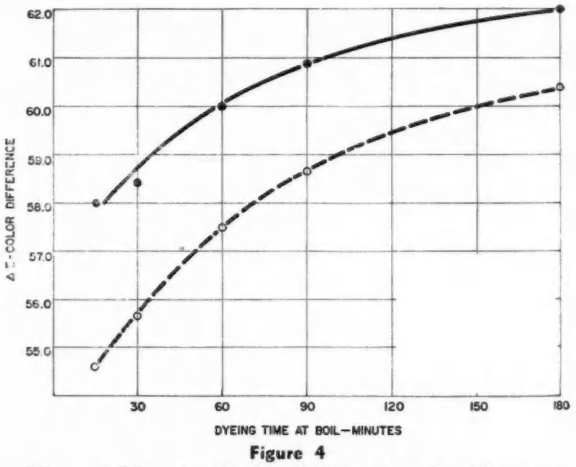

Figure 4

Effect of 5.0% (owf) of surfactants on color difference of Arnel. (Solid line represents Igepon T; broken line indicates nonionic).

Dyed with:
0.8% Eastman Fast Blue BGLF
0.2% CI Disperse Red 35
0.2% Interchem Acetate Yellow HDLF-40
0.5% CI Basic Blue 22
0.2% Sevron Red 4G
0.2% Sevron Orange G

Heat increases the solubility and dissociation of this compound. During dyeing the equilibrium is shifted by removal of the dye cation from the bath. A dissociation of the compound occurs, thus making available additional dye cations for the fiber. Without discriminate selection of the anionic surfactant used, it is postulated that compounds can be formed which have decreased solubility, are not easily dissociated, and in which agglomeration can occur to give a precipitate. Additional investigations are required to permit understanding of the mechanism involved.

pH EFFECT——The effect of pH on cationic and disperse dyes in a one-bath system was investigated. The dyebaths contained 0.5% (owf) of Igepon T and were adjusted to pH's of 4.5, 6.0, 6.5, 7.0 and 8.0 with acetic acid and TSPP. A tertiary disperse dye mixture containing Eastman Blue

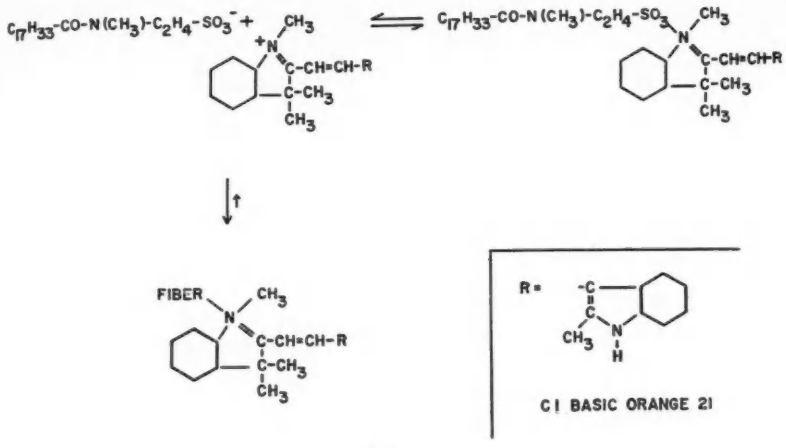

Figure 5
Mechanism of surfactant-cationic dye association

BGLF, CI Disperse Red 35, and Interchem Acetate Yellow HDLF-40 was used in all baths for dyeing the Arnel. One percent (owf) of nine representative cationic dyes were checked individually in the presence of the disperse dye mixture at the various pH's. A 50/50 Arnel/Orlon combination test fabric was used in each dyeing. The dyeings were run two hours at a boil. The following conclusions were drawn from this work:

A) Neutral-dyeing cationic dyes at pH 6.0-7.0 show duplicable cast and shade build up which is equivalent to their true shade and depth.
B) As expected, loss in shade depth occurred with the acid-dyeing cationic dye as the pH became more alkaline.
C) Retarding of some of the cationic dyes occurs at a pH of 8.0.
D) Less staining of the Orlon by disperse dyes is noted at pH of 6.0-7.0.

The pH range of 6.0-7.5 has proved to be practical for dyeing the Arnel/Orlon blend with disperse and selected cationic dyes in the same bath; however, for processing in a commercial dyehouse, it has been found desirable to minimize pH variations due to water supply, etc, by the use of pH buffering assistants. The use of 1% monosodium phosphate and 2% disodium phosphate (owf) will control the pH between 6.8 and 7.0 in normal dyebath liquor ratios.

ACCELERANTS——Several accelerants for Arnel were evaluated to determine their effect upon the one-bath process. Laboratory and plant trials were run using mixtures of disperse and cationic dyes to give a navy shade. The effect of accelerants on the individual neutral-dyeing cationic dyes was observed in the laboratory. The following types of accelerants were evaluated:

o-phenyl phenol
methyl salicylate
butyl benzoate
alkyl phosphate
acetyl triethyl citrate
dimethyl terephthalate—benzanilide combination.

Visual observations and dyebath filtration test confirmed that, with all the accelerants, the dyebaths were stable. However, of all the commercial carriers evaluated, only Latyl Carrier A was found to be satisfactory for the one-bath procedure. The main deficiency of the other carriers was that they retarded too severely the exhaustion of the cationic dyes onto the Orlon. The retarding effect of these carriers was probably due to the anionic nature of the emulsifying agents used with the accelerant. When a carrier is used, less staining of the Orlon with disperse dyes occurs. This results in cleaner and brighter cross-dyed shades.

When using Latyl Carrier A in the one-bath procedure, the amount of Igepon T in the dyebath should be increased to 1.0%. Latyl Carrier A in concentrations of 0.5-1.0 g/l is added to the bath at 120°F after the dyes have been added. In heavy shades the use of Latyl Carrier A will permit a 20-30% reduction in the amount of disperse dye required.

Laboratory and plant dyeing trials have shown the necessity of adding the cationic and disperse dyes separately to the dyebath. Sufficient time should be allowed for the cationic dyes to be thoroughly diluted in the dyebath before the addition of the disperse dyes. Any tendency for precipitation or agglomeration to occur is most prevalent when cationic and disperse dyes are mixed together in a concentrated condition.

TABLE V
One-bath dyeing procedure for Arnel/Orlon

1) Scour fabric 20 minutes at 160°F with 1.0% nonionic or anionic surfactant and 1.0% TSPP. Rinse.
2) In a new bath at 120°F add 0.5%—1.0% Igepon T, 1.0% monosodium phosphate and 2.0% disodium phosphate. Run five to 10 minutes.
3) Dissolve cationic dyes in methyl alcohol and hot water. Add dissolved dyes to bath and run five to 10 minutes.
4) Dissolve and strain disperse dyes. Add dissolved dyes to bath and run five minutes. If required, add 0.5—1.0 g/l Latyl Carrier A.
5) Raise temperature evenly to 180°F over 30 minutes. Raise temperature evenly from 180°F to a boil over 30 minutes.
6) Run 60-90 minutes at a boil before sampling.
7) Cool back to 180°F before making adds.
8) When on shade, open the doors to the machine and cool the bath slowly by radiation to 160°F.
9) Add inhibitor during cooling cycle at 180°—190°F.
10) Gradually cool bath down and rinse with warm and cool water.
11) Scour heavy shades 20 minutes at 130°F with 1.0% nonionic surfactant. Rinse.
12) Apply in final rinse bath for 20 minutes at 120°F 0.5%—3.0% cationic softener. Offload.

From this work, a practical one-bath procedure for dyeing Arnel/Orlon blends has been developed which is based on the following principles:

1) Use of selected neutral-dyeing cationic dyes.
2) Use of a selected anionic surfactant, Igepon T.
3) Control of pH at 6.5-7.0.
4) Separate addition and dilution of cationic and disperse dyes.

The one-bath procedure shown in Table V is used to dye union and cross-dyed shades on Arnel/Orlon tubular knit fabrics.

Several hundred thousands of pounds of Arnel/Orlon jersey and high bulk yarns have been successfully dyed by American & Efird Finishing Co and Banner Yarn Dyeing Corp using these principles.

PART II OF THIS PAPER WILL APPEAR IN THE FEBRUARY 22 ISSUE. THE FINAL INSTALLMENT (PART III) WILL APPEAR IN THE MARCH 7 ISSUE.

38th National Convention———

DYEING BLENDS OF DACRON POLYESTER FIBER AND COTTON BY THE DU PONT THERMOSOL PROCESS*

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The Thermosol Process is a continuous method of dyeing whereby dye fixation and penetration are accomplished with dry heat. It is particularly effective in the application of disperse dyes to Dacron polyester fiber and its blends with cellulosic fibers. In dyeing this important blend, selected disperse dyes are used to dye the Dacron by the Thermosol Process and, where necessary, vat dyes are applied to the cotton by conventional procedures. Fabrics dyed in this manner are generally equal in fastness to vat-dyed cotton and will meet "wash-and-wear" fastness requirements.

For many pastel shades, it may be possible to dye only the cotton portion of the blend with vat dyes applied by the pad-steam process; for light-to-medium shades, dyeing only the Dacron by the Thermosol Process may suffice. However, in heavy shades, it is necessary to dye both fibers. Recent laboratory work indicates that it may be possible to dye both the Dacron and the cotton in a one-pass operation using disperse dyes and fiber-reactive dyes, both of which can be applied to the respective fibers with dry heat.

Blends of Dacron type 64 polyester fiber and rayon can also be dyed advantageously by the Thermosol Process.

Fixation and penetration of disperse dyes applied by the Thermosol Process is very rapid. Dye utilization is good and compares favorably with that obtained by conventional aqueous procedures.

INTRODUCTION

SHORTLY after the introduction of Dacron polyester fiber, a continuous procedure for its dyeing was announced. This procedure, called the Du Pont Thermosol Process, was rather unique in that dry heat was used to fix the dye on the fiber (*1,2, 3,4*). To be sure, this new method of dyeing was not adopted immediately by the trade, but there were good reasons for the apparent lack of enthusiasm. First of all, the Thermosol method involved continuous dyeing, which meant that fairly large yardages of a given shade would have to be processed to make continuous operation economically attractive. In these early formative years, Dacron was still in the process of establishing itself in the textile industry and with the consumer; hence, large yardages of any given shade were rare. Secondly, one of the first important uses for Dacron was in blends with wool, a combination which did not readily lend itself to the high temperatures involved in the Thermosol Process because of the possibility of damage and yellowing of the wool.

The Thermosol Process, therefore, remained a laboratory curiosity until a few years ago when the relatively new Dacron found an ideal partner in one of the oldest fibers known—cotton. The intimate blend of these two fibers, usually in the proportion of 65% Dacron and 35% cotton, was an immediate success and gained rapid consumer acceptance. Soon the yardages of this important blend reached such proportions where a continuous operation was warranted, and the Du Pont Thermosol Process was revived.

This paper deals primarily with practical applications of the Thermosol Process in dyeing blend fabrics of Dacron and cotton. Most of the discussion will be based upon actual mill experience and will emphasize the fact that the Thermosol Process of dyeing is no longer a laboratory curiosity but is, in fact, a commercial reality.

Unless otherwise specified, all references to the Dacron in the blended fabric signify Dacron Type 54 polyester fiber. At the present time the relatively new Dacron Type 64 is used primarily in blends with wool and rayon. A short discussion regarding the dyeing of the blend of Dacron Type 64 with rayon will also be included.

CONTINUOUS DYEING OF FABRICS OF DACRON AND COTTON

PREPARATION———Proper preparation of the fabric is an important requisite in any continuous dyeing operation. In the case of Dacron and cotton, the fabric must be absolutely clean since residual oils or dirt would be set into the fabric by the Thermosol treatment. Furthermore, the fabric must be wet out instantaneously and uniformly in order to insure adequate absorption at the padder.

Several preparation routes are available. The one selected will depend, of course, upon the ease with which it fits into the continuity of processing in a particular mill. A typical procedure which has given satisfactory results on commercial yardages is given in Table I.

Since in many instances little or no starch sizing is used on yarns of Dacron and cotton, it may be possible to eliminate the desizing operation entirely and begin the preparation by mercerizing in the greige. This alternative is especially beneficial when

TABLE I
Typical preparation procedure for fabrics of Dacron and cotton

1) Desize
2) Rope scour or modified* progressive jig boil off
3) Mercerize
4) Dry
5) Singe
6) Open-width bleach (if necessary)
7) Frame dry to greige width.

*4.0% soda ash and 1.0% surfactant or 2.0% caustic soda if dwell time is not over three hours.

*Presented October 9, 1959 during the 38th National Convention, held at Washington, DC

the fabric contains waxes, since they are efficiently removed in the mercerizer.

It should be noted that singeing can be done before dyeing by the Thermosol Process whereas it is usually avoided prior to aqueous dyeing. Singeing may produce tiny melt balls on the surface of the fabric, and heat differences across the fabric. In aqueous dyeing, the dye is free to migrate to the more dyeable portions; the melt balls dye deeper and give the fabric a speckled appearance and the non-uniform heating may show up as shading across the piece. With the Themosol process, however, the dye is padded onto the material and dyeing takes place more or less "in situ" so that differences in dyeability from one part of the fabric to another are effectively covered.

DYEING BY THE DU PONT THERMOSOL PROCESS———

General Discussion. In the Du Pont Thermosol Process, the fabric is padded with a solution or dispersion of the dye, dried and finally subjected to a heat treatment at temperatures ranging from 400° to 425°F for 45 to 90 seconds. Under the influence of the dry heat, the originally padded dye penetrates rapidly and completely into the fiber. (Fig 1).

Although many of the newer synthetic fibers can be dyed with various classes of dye by this procedure, the most outstanding results have been obtained in the application of disperse dyes to the Dacron in blend fabrics of Dacron and cotton.

The Thermosol Process offers several distinct advantages over aqueous batch-dyeing procedures. These are:

1) It is a continuous process, hence large yardages can be dyed economically.
2) No carrier is required, hence the danger of spotting and the adverse influence of some carriers on light fastness are eliminated.
3) Dye utilization is excellent.
4) Fabric is processed in open width, eliminating rope marks.
5) Heat setting and dyeing occur simultaneously.
6) Dyeability is not affected by preheat setting.

Each of the three basic steps, ie, padding, drying and heating to fix the dye, must be carried out carefully and under properly controlled conditions for optimum results.

In the preparation of the padding bath, care should be taken to insure complete dispersion of the dyes and to avoid specking. A suitable procedure follows:

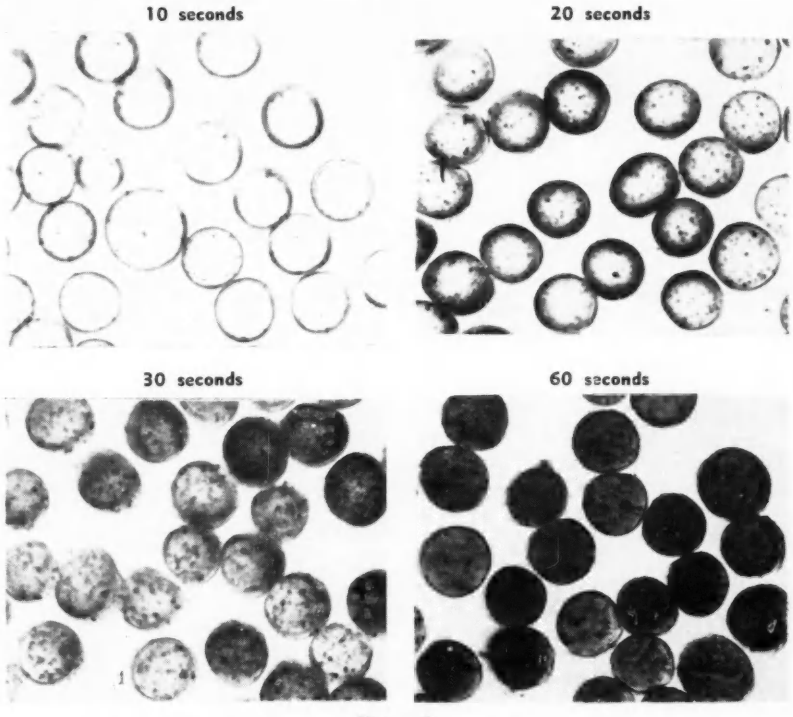

Figure 1
Penetration and fixation of a disperse dye applied by the Thermasol Process
Temperature = 400°F

1) In approximately one-half the desired volume of hot water (190°-200°F) add 1.0 ounce per gallon Compound #8S (based on total volume).
2) Sprinkle in the desired amount of dye with vigorous hand stirring or mechanical mixing.
3) Preferably pass dispersion through an Eppenbach or colloid mill.
4) Add 0.15 ounce per gallon alginate thickener (to improve wet pickup and control migration) and 0.2 ounce per gallon sodium hydrocarbonsulfonate (to insure wetting).
5) Dilute to volume.
6) Pad at 120° to 140°F.

As is the case in the continuous application of vat dyes to cotton, the next step, drying, is quite critical. The disperse dyes, like some vat pigments, have a tendency to migrate in the drying operation and every precaution should be taken to avoid this difficulty. The use of a radiant-heat predryer for partial drying is beneficial in controlling migration, but it is not considered absolutely necessary. The presence of the thickener and a minimum of wetting agent will also help in controlling migration, but uniform heat and balanced air circulation in the drying chamber are of utmost importance; otherwise, cross-shading or back-to-face shading may result. The temperature of drying should be between 250° and 300°F.

After drying, the fabric is preferably conveyed to the Thermosol unit over a clip frame. The Thermosol unit should be a roller-type oven in which temperatures of 400° to 450°F are obtainable. Optimum time of exposure to the dry heat will vary somewhat depending upon the equipment, depth of shade, and weight of the fabric being processed but, in general, about 60 seconds at 400° to 425°F is sufficient for complete fixation and penetration of dye into the Dacron (5). Here again, balanced air flow and consistent heat throughout the oven are important requisites for uniform shade development.

The time and temperature requirements for dye fixation in the Thermosol unit are also sufficient to accomplish heat setting at the same time. However, since the fabric is unrestrained, especially in the filling direction, it is free to shrink to its fullest capacity; and width control and the development of creases, particularly in lightweight fabrics, may present some problems in some constructions. The best solution to these problems developed to date is to frame the fabric to greige width or perhaps an inch beyond between the drying oven and the Thermosol unit. Thus, the usefulness of a frame between the two ovens is readily apparent. To minimize crease forma-

TABLE II
Du Pont disperse dyes recommended for application by the Thermosol Process

Latyl Yellow YL* (Pr 625)	Latyl Violet 2R
" Yellow 3G	" Bordeaux B
" Yellow RC	" Blue 4R
" Yellow 4RL (Pr 583)	" Blue 2R
" Orange NST	" Blue BCN
" Orange 3R	" Blue LS
" Cerise B*	" Brill Blue 2G
" Red GFS	" Brill Blue BG
Celanthrene Violet BGF (CI Disperse Blue 8)	

*These dyes may cause specking in powder form unless thoroughly milled and screened. They are available as paste types which are essentially free from this deficiency.

tion, it is desirable to have driven rolls or to install bow bars in the Thermosol unit.

The equipment used for dyeing by the Thermosol Process will undoubtedly be recognized as the usual resin-curing ranges which are readily available in many modern mills. Indeed, most of the successful commercial dyeings made to date, which in some cases exceeded 10,000 yards, have been processed on just such equipment.

The short time of exposure required for complete Thermosol fixation of the dyes insures a high rate of production. This would be dependent upon the capacity of the Thermosol unit and the speed at which it operates, but a rate of 60 to 80 yards per minute is considered normal.

Dyes. The most important class of dyes applied to Dacron polyester fiber by the Thermosol process is undoubtedly the disperse types. Selected vat dyes can also be applied either in pigment form or as the vat acid but, due to the limited selection and difficulty with shade and union control, these have not been widely adopted (*1,3,6,7*).

Disperse dyes offer a wide range of shades, good color value and a high degree of fastness when applied to Dacron polyester fiber by the Thermosol process. To exercise optimum shade control and to prevent contamination of the unit, only those dyes which exhibit minimum vaporization in the Thermosol operation should be employed. Table II lists disperse dyes manufactured by the Du Pont Company, which have been found suitable in commerical operation for application by the Thermosol methods.

It has long been recognized that small residual quantities of some carriers exhibit an adverse influence on the lightfastness of certain disperse dyes. Since no carrier is employed in the Thermosol Process, these dyes, which often offer an attractive economic advantage over those less affected by residual carriers, can be used.

Recent mill experience has substantiated earlier laboratory data which showed that the degree of dye utilization in the Thermosol operation was good and compared quite favorably with that of aqueous carrier dyeing (5). Generally, between 75% and 90% of the originally padded dye is retained by the Dacron component of the blended fabric.

Selected vat dyes, when used in pigment or vat acid form, can be applied to Dacron by the Thermosol Process. To achieve optimum dye fixation, a full 90 seconds at 410° to 425°F is required (5). They offer an advantage in that only one padding operation is required if the same dye produces a fairly good union shade on both the Dacron and the cotton portions of the blend. In the Thermosol operation, a portion of the vat-dye pigment is fixed on the Dacron and the remaining portion is then fixed on the cotton by conventional continuous or batch procedures. The vat types must therefore be selected on the basis of utilization on the Dacron and union effect on both fibers.

It should be noted that some vat dyes which initially show a good union effect may be only partially fixed on the Dacron by the Thermosol operation. Accordingly, the caustic soda and reducing agent required to fix the vat dye on the cotton may strip a substantial portion of the dye from the Dacron, thereby resulting in difficult shade control and poor balance between the two fibers, particularly in heavier shades (5). For this reason, where the use of vat dyes is contemplated, the concentration should be kept to a minimum. For the most part, vat dyes should be limited to the production of light-to-medium shades. Since these shades can be made more economically by other means, it is not expected that vat dyes will be used to any great extent for dyeing Dacron by the Thermosol process. Suitable vat dyes which yield a good union effect and fair-to-good color value are listed in Table III (6).

Practical Application of the Du Pont Thermosol Process. Basically, the continuous coloration of blends of Dacron and cotton is accomplished with disperse dyes for the Dacron, and vat, sulfur, naphthol, fiber-reactive or direct dyes for the cellulosic fiber. Some of these combinations, when properly applied, generally produce the high degree of fastness required for the "wash-and-wear" performance for which many such fabrics are intended.

The dyes can be applied in a variety of ways to produce the desired results. The method of application selected will depend upon several factors, including the depth of shade, equipment, and ultimate fastness desired. Some of the possible combinations will be discussed below.

Disperse Dyes and Vat Dyes Applied Separately. In the production of medium-to-heavy shades, it will be necessary to dye both components of the blend. By far the most suitable

TABLE III
Selected vat dyes suitable for application by the Thermosol Process

50 g/l of dye	*Union effect*	*—Dacron vs cotton— Depth of shade*	*Direction of shade*	*Lightfastness Fade-Ometer hours to appreciable break Blend of Dacron and cotton*
CI Vat Yellow 16	fair	similar	3R	20
CI Vat Yellow 3	good	4S	4G	80
CI Vat Yellow 21	excellent	similar	4G	20
CI Vat Orange 9	good	3W	4R	160
CI Vat Orange 5	good	3S	similar	20
CI Vat Brown 40	good	similar	4Bl	160
—	good	3W	similar	160
CI Vat Violet 12	good	similar	4Bl D	80
CI Vat Green 3	excellent	similar	similar	80
CI Vat Black 25	good	similar	5-4R	160
CI Vat Black 14	good	similar	4Bl D	80

Legend: Bl—bluer
D—duller
R—redder
S—stronger
W—weaker

method to accomplish this to date has been to apply disperse dyes to the Dacron portion by the Thermosol Process and then dye the cotton by conventional batch or continuous procedures. This approach offers two important advantages: 1) the cotton-dyeing procedure constitutes an effective scour for removing disperse-dye stain from the cotton, particularly in batch procedures, and 2) maximum shade control can be maintained. Of course, this system involves two separate and distinct padding and dyeing operations—one for the Dacron, the other for the cotton, but for high-quality work the results are certainly worth the effort.

Disperse Dyes and Vat Dyes Applied Together. In this procedure disperse and vat dyes are padded together from the same bath; the fabric is dried as usual and subjected to the Thermosol treatment. The one outstanding advantage in this approach is, of course, that only one padding operation is required. However, shade control can be a problem since, as mentioned previously, some of the vat dye will fix on the Dacron during the Thermosol step; so, in effect, the shade on the synthetic component is made with a combination of vat pigments and disperse dyes. It will be necessary, therefore, to select vat dyes which, while supplementing the shade of the disperse dyes on the Dacron, will, upon reduction, also produce the desired union effect on the cotton portion. Also to be considered is the stripping action exerted by the reducing chemicals on the vat dye on the Dacron, which will make it difficult to maintain the shade on one fiber while dyeing the other, thereby complicating shade control.

In order to utilize this approach effectively, it will be necessary to match the shade carefully with disperse and selected vat dyes, taking into consideration all of the factors discussed above. Once the shade is established, it should repeat satisfactorily under similar processing conditions.

Disperse Dyes Only. In dyeing very light pastel shades, it has been the practice in many mills to dye only the minor cotton portion of the blend with vat dyes applied by the Du Pont pad-steam process. This procedure has been particularly effective on lightweight shirting fabrics, batistes and broadcloth.

For certain light - to - medium shades, such as beige, turquoise, maize and pinks, dyeing only the cotton may not produce acceptable coverage. In such instances, satisfactory solidity of shade can be obtained by dyeing only the Dacron with disperse dyes applied by the Thermosol process. Of course, the major advantage of this approach is that the fabric can be dyed in a simple one-pass operation. Fastness to light and washing of those fabrics where only the Dacron is dyed has been found to be quite satisfactory.

TABLE IV
Latyl disperse dyes compatible with the alkaline fiber-reactive system

Yellow 3G*
Yellow YL*
Yellow 4RL*
Orange 3R*
Bordeaux B
Blue 2R
Blue BCN*
Brilliant Blue BG*
Brilliant Blue 2G*

*Exhibit optimum utilization from the alkaline fiber-reactive system.

Disperse Dyes and Fiber-Reactive Dyes. The simultaneous application of disperse and fiber-reactive dyes offers another possibility for dyeing both the Dacron and the cotton in one operation. In this application, the disperse and fiber-reactive dyes are applied to their respective fibers by the combined Thermosol and thermofix procedures (*8*).

In the thermofix method, the fiber-reactive dyes are dissolved in water containing up to 200 g/l urea and 20 g/l soda ash[1]. The fabric is then padded, dried and subjected to a short exposure to dry heat whereupon the dye is fixed to the cellulose (*9,10*). By incorporating the disperse dyes in the same pad bath, it should be possible to dye both fibers in one heat-fixing operation.

Many encouraging results have been obtained in the laboratory and in small-scale mill dyeing, especially in light-to-medium shades. Certainly this system of dyeing offers the greatest potential for dyeing both fibers in blends of Dacron with cotton or rayon in one simple operation.

However, there are several problems connected with this approach. The alkaline pad bath required for the application of the fiber-reactive dyes causes certain disperse dyes to precipitate and speck the cloth badly; hence, the disperse dyes must be carefully selected. The use of special dispersing agents helps in minimizing this problem but does not overcome it entirely. Those Latyl disperse dyes which have been found to be suitable for use in the alkaline fiber-reactive system are listed in Table IV.

Many of the disperse dyes, although compatible with the alkaline fiber-reactive system, yield shades which are considerably weaker than those obtainable with a similar concentration of dye applied by the regular Thermosol Process. To take advantage of the one-pass dyeing operation, it may therefore be necessary to sacrifice some of the disperse dye efficiency. The relative economics of the one-step process versus the two-step method employing disperse and vat dyes would have to be carefully evaluated.

Because of the relatively low wet pickup of blend fabrics of Dacron and cotton, the pad baths required for the production of heavy shades are necessarily quite concentrated. It has been noted that, in high concentrations, the solubility of certain fiber-reactive dyes is adversely affected, especially in the presence of high concentrations of disperse dye. As a result, the shade produced on the cotton portion of the blend is weak. For example, a good black shade can be made by applying the disperse and fiber-reactive dyes in separate but successive Thermosol and thermofix operations; when both dyes are padded from the same bath, a charcoal shade results. The disperse dyes are somewhat weaker on the Dacron because of the alkalinity of the pad bath and the fiber-reactive dyes are much weaker on the cotton, presumably because of poor solubility or because of interaction with the disperse dye and/or dispersing agent(s). (Fig 2).

The problems discussed above are not insurmountable and it is expected that further research will lead to more widespread use of this procedure.

DYEING BLENDS OF DACRON TYPE 64 POLYESTER FIBER AND RAYON——Although the relatively new Dacron Type 64 is not used in blends with cotton, considerable quantities are being blended with rayon for the production of suitings and work clothing. It is expected that the volume of such fabrics will soon reach a sufficiently high level to warrant continuous operation.

The dyes and procedures previously recommended for dyeing blends of

[1]The use of an acid binding agent, such as soda ash, in the application of cyanuric-chloride-type fiber-reactive dyes is covered in U S Patents 2,892,670 and 2,892,671 (assigned to Ciba).

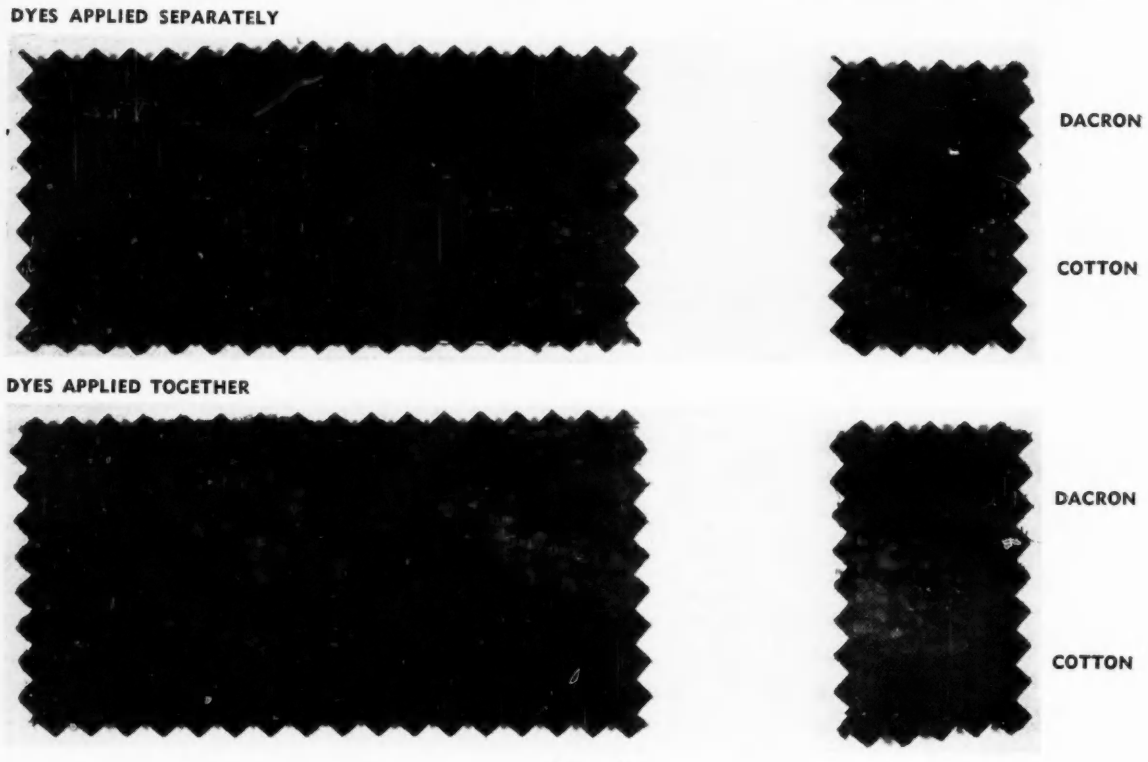

Figure 2
Application of disperse and fiber-reactive dyes
90 seconds at 400°F

Dacron Type 54 and cotton by the Thermosol Process can also be used for blends of Dacron Type 64 and rayon.

Data obtained from small-scale mill trials shows that utilization of disperse dyes on Dacron Type 64 is fully equal to that obtained on Dacron Type 54.

CONCLUSIONS

Experience with numerous full-scale mill trials indicates that blends of Dacron and cotton can be dyed efficiently and economically by the Du Pont Thermosol Process. Selection of the disperse and/or vat dyes used in this operation is critical, but dyes suitable for making a full range of shades at reasonable cost are available.

The most widely used procedure is to dye the Dacron first with disperse dyes by the Thermosol Process and then dye the cotton with vat dyes applied by conventional batch or continuous procedures. For certain light-to-medium shades, it may be possible to dye only the Dacron with disperse dyes applied by the Thermosol Process. Selected vat dyes may also be used for both fibers.

An encouraging approach for dyeing both the Dacron and cotton in one heat-fixing operation includes the co-application of disperse and fiber-reactive dyes. However, this procedure is still in the development stage and further refinement is required.

Blends of the relatively new Dacron Type 64 and rayon can be dyed with the same dyes and procedures used for blends of Dacron Type 54 and cotton.

Full commercialization of the Du Pont Thermosol Process is a commercial reality in several mills. The use of this dyeing procedure should spread rapidly as increased quantities of fabrics become available and the need for long yardages of a given shade increases.

ACKNOWLEDGMENT

The authors respectfully acknowledge the assistance of E N Sarantis for his contribution on the simultaneous application of disperse and fiber-reactive dyes.

TRADEMARKS

The following are registered trademarks of the E I du Pont de Nemours & Co, Inc: Celanthrene, Dacron and Latyl.

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38th National Convention———

RECENT ADVANCES IN THE PRESSURE BEAM DYEING OF SYNTHETIC FABRICS*

WALTER J NEWCOMB
Gaston County Dyeing Machine Company

and

GEORGE C WARD
Celanese Fibers Company
A Division of
Celanese Corporation of America

Pressure beam-dyeing machines have been available for the last decade. The usefulness of this type of machine has been consistently demonstrated for the coloring of certain types of fabrics, among which are acetate tricot, Arnel tricot, nylon tricot, Arnel and nylon marquisette and ninons, filament Dacron fabrics, blends of polyester fibers and cotton and blends of polyester fibers with wool.

Recent mechanical advances in pressure beam-dyeing machines include automatic heating and cooling, automatic pressure and flow control, automatic static pressure chamber, a closed pressurized expansion tank, a running wash system and an automatic beam revolving device. General methods are given for the processing of typical standard fabrics, including those of blends of synthetic and natural fibers. Suggestions are made for getting level dyeing, satisfactory fastness and proper setting of the fabric.

INTRODUCTION

DYERS have long realized the need for high-temperature dyeing of the new synthetic fibers. Gaston County Dyeing Machine Co decided to make a very careful study of this situation in cooperation with fiber manufacturers and dyers in order to improve machines and methods used to dye fabrics made from synthetic, natural, and blended fibers. Part of the work reported in this paper was carried out in cooperation with the Dyeing & Finishing Laboratories of Celanese Fibers Co and E I du Pont de Nemours & Co, Inc.

DISCUSSION

There are only two common methods of dyeing—the fabric either moves through the dye liquor or the dye liquor is forced through the fabric. It was apparent that, to make a pressure piece-dye kettle, such as a dyebeck or jig, the cost would be prohibitive due to the construction of this type machine. It was also realized that there was a strong possibility that creases and rope marks would form on a pressurized dye beck, which would be practically impossible to remove once they are set at a temperature above the boil. Likewise, there was also the strong possibility in a pressure jig that creases would form, and unknown to the operator, would seriously damage a complete roll of fabric.

DuPont attacked this problem by development of the Barotor, which is fully covered in a paper by Cole (*1*). Some difficulties have been experienced on this machine, which are well known to practical dyers. Thus, the next plausible step was to design a machine to circulate the dye liquor through a roll of fabric, under pressure. These were described in several patents (*2,3,4,5*). This is basically the same principle as package yarn dyeing or yarn beam dyeing.

In America, this was done by the use of one large beam, but a number of Colorfix machines have been introduced in Europe, made under the Bellman patent (*6*). In this machine, three to six small-diameter beams are introduced into the autoclave. American experience has shown that the single large-diameter beam is more adaptable to our procedures. It has been found that, in package dyeing of yarn, more satisfactory results are obtained on a 1-5/8" tube than on a 5/8" tube. This principle likewise follows in dyeing fabric on beams. Beams have been fabricated here with a diameter of as much as four feet. On a beam of this size, fabric of reasonably open texture may be dyed in batches up to 3500 yards. On the contrary, using the Colorfix machine, the largest size roll used in Europe contains about 300 yards.

In many cases in Europe, single pieces of quite tightly woven fabric, such as taffeta or satin, are dyed on each beam, but only 60-90 yards are put on each beam. Extreme care must be exercised to insure that the fabric is wound with the same degree of density, otherwise there will be considerable variation in shade from beam to beam.

The basic design of the American machine is a horizontal kier with the beam held in a horizontal position. Figure 1 illustrates the largest-size, commonly used beam. Figures 2 and 3 are views of the entire assembly. The machine has reverse flow inside-out and also outside-in. It has been found that most fabrics can be dyed with inside-out flow only; however, it may be necessary to reverse the flow on some tightly woven fabrics. New features which have greatly improved the beam-dyeing operation are as follows:

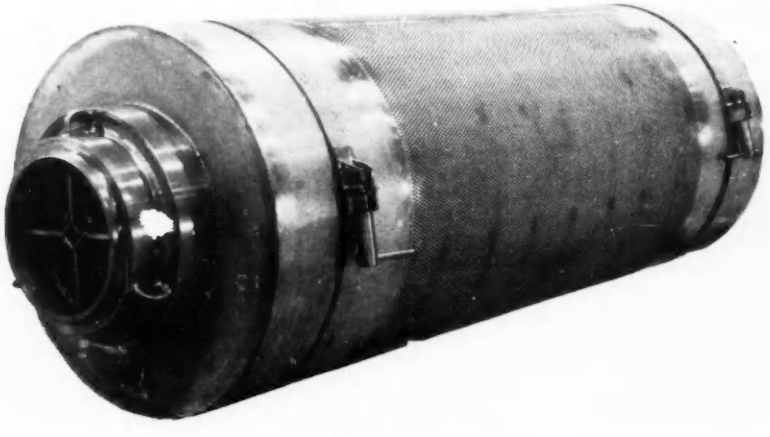

Figure 1
Open-width cloth dye beam with beam bands (30" diameter)

*Presented on October 9, 1959 during the 38th National Convention, held in Washington, DC

1) Automatic heating and cooling.
2) Automatic pressure and flow control.
3) Automatic static-pressure chamber.
4) Closed pressurized expansion tank.
5) Running wash system.
6) The automatic beam-revolving device.

AUTOMATIC HEATING AND COOLING———This is done through a heat exchanger, which is controlled through the control panel to give predetermined rate of rise and also a predetermined holding time. The temperature can be held within 1°F, which is very helpful in dyeing some fabrics and also some critical shades. The controller maintains preset temperatures at all times through the exchanger.

AUTOMATIC PRESSURE AND FLOW CONTROL———The desired dyeing pressure is preset on the control panel and is automatically controlled and maintained throughout the dyeing cycle. The pressure and flow may vary with different size batches, and also with fabric construction. The flow control valve is always closed when the dye pump is not running and it also closes during reversal periods. The valve also closes before the flow is reversed from outside-in to inside-out, or vice versa. After the flow is reversed, the valve opens slowly. This eliminates sudden surges which disturb the position of the fabric on the dye beam. A flow recorder can be installed when desired. This will give a permanent record of each dye lot.

AUTOMATIC STATIC-PRESSURE CHAMBER———With the static-pressure chamber, compressed air can be injected into the vessel. This acts as a cushion which allows the dye pump to maintain a constant flow.

CLOSED PRESSURIZED EXPANSION TANK———The closed expansion tank can be used with the compressed air cushion for pumping and, also with this tank, the entire dye charge can be used when dyeing above the boil. With the open tank, when dyeing above the boil, the expansion must be cut off, thereby losing the dye liquor in the tank.

RUNNING WASH SYSTEM——— Clean water is fed to the dye pump from the expansion tank, through a large pipe line. The clean water is forced through the dye beam and then exhausted to the drain sewer. This gives a much cleaner and faster wash.

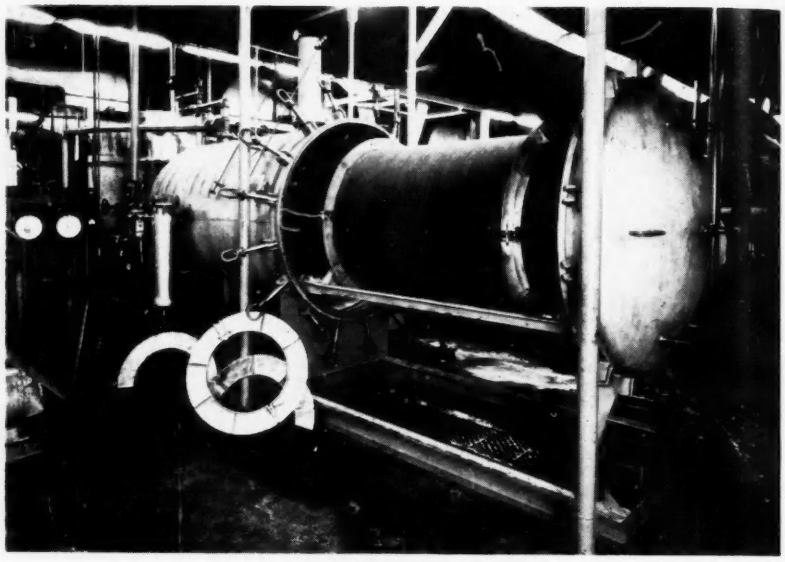

Figure 2

Woven fabrics—beam-dyeing machine—front view

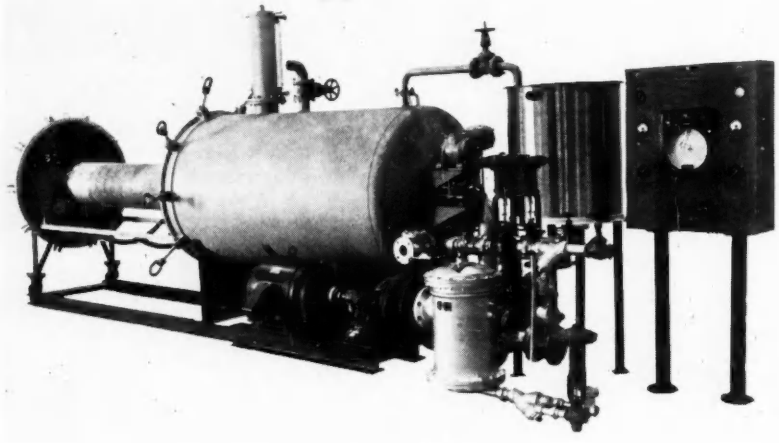

Figure 3

Woven fabrics—beam-dyeing machine—rear view

AUTOMATIC BEAM-REVOLVING DEVICE———The beam is slowly revolved by a constant-speed driving mechanism. This aids penetration, gives a slight agitation during the scouring and dyeing cycle, and will tend to minimize shading and help produce level dyeing from end to end. It will also move or help to dissipate any air bubbles that may form between layers of fabric. These bubbles sometimes cause light spots in fabric.

TYPICAL PREPARATION PROCEDURES

In order to get a level dyeing, one must be sure that the fabric and the beam are properly prepared. Figure 4 illustrates a typical installation for beam preparation. The following procedures are suggested:

1) Fabrics with a high potential for shrinkage should first be boiled off. This is necessary to take out the initial shrinkage before dyeing, to give a beam which will maintain a constant density throughout the dyeing process, and to give a final fabric of good dimensional stability.

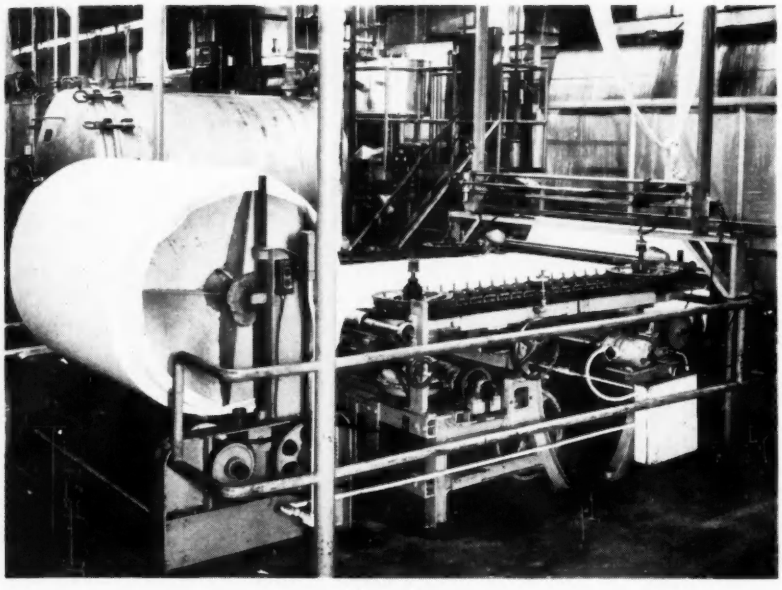

Figure 4

2) The fabric should, if possible, be batched wet or damp, since the fabric seems to lay better in this state.
3) It has been found that more satisfactory results have been obtained if a cotton leader is tightly wrapped around the beam before the batching of the fabric to be dyed.
4) Care should be taken in batching so as to place the fabric evenly over the perforations and free from creases and folds.
5) The fabric should overlap the perforations on each end one and one-half to two inches for woven fabrics, and two to three inches for knitted fabrics.

Where a dyer is running fabrics of a wide variety of widths, he will find it advantageous to have on hand beams with perforations to suit each width of fabric. The blocking off of perforations for narrow-width fabric is not recommended. However, the use of a stainless-steel sleeve, in lieu of a beam of the proper width, has been used with success.

TYPICAL DYEING PROCEDURES

ARNEL TRIACETATE TRICOT ——The first fabric successfully dyed on pressure beam-dyeing machines was Arnel triacetate tricot (*7,8*). Many millions of pounds of Arnel tricot have been dyed into a complete range of shades, resulting in a fabric of good dimensional stability, good antistatic properties, completely heat set and possessing ease-of-care properties. In order to get heat-setting properties, the Arnel tricot is treated for 30-45 minutes at 250°F, but normally dyeing is not conducted above 220°F, since many of the dyestuffs tend to break down at higher temperatures.

POLYESTER STAPLE FIBERS AND COTTON (ALSO RAYON) (*9,10*)——These fabrics are prepared for dyeing and finishing as all-cotton fabrics except for a few modifications to control the pilling tendencies of polyester fibers. They are usually scoured and heat set to give dimensional stability. These blends with cotton can be union and cross dyed satisfactorily using disperse dyes on the polyester fibers and direct dyes on the cotton. In a similar manner, fabrics made of blends of polyester and rayon can be union or cross dyed.

BLENDS OF POLYESTER FIBERS WITH NYLON——These fabrics can be dyed using disperse dyes on the polyester fibers and shading over the nylon with acid dyes. They can be dyed either by a one-bath or a two-bath method. The usual dyeing procedure will take three to four hours and is normally conducted at around 230°F.

BLENDS OF POLYESTER FIBERS AND WOOL——Commercial quantities of Dacron/wool fabrics are being dyed on the pressure beam-dyeing machine these days (*11*). Such fabrics are dyed with disperse dyes for the polyester fibers and either level-dyeing chrome or neutral-dyeing premetalized acid dyes for the wool. For heavy shades, most of this fabric is probably being dyed by a two-bath procedure, but for light and medium ones the one-bath method is employed, ie, disperse dyes and neutral acid colors are applied simultaneously. An advantage of pressure dyeing Dacron/wool fabrics is the fact that a larger amount of the disperse dye is picked up by the Dacron, thus less staining of the disperse color occurs on the wool. This is important since disperse dyes have notoriously poor lightfastness on wool. It is well known that wool loses some of its strength when exposed to high temperatures for long. Therefore, good laboratory recipes must be available so that the dyeing process is not continued for extensive periods. Under controlled conditions, medium and heavy shades are obtained on this blend within a two-hour dyeing cycle.

Usually this blend is not dyed above 230°F. Even though the wool is tendered, the overall strength of the fabric is adequately taken care of by higher strength of the polyester fiber and the ultimate result is a better-appearing and better-performing fabric than would be obtained if the fabric was dyed on a dye beck for a much longer time and with the use of a carrier or accelerant.

SHEER FABRICS OF 100% CONTINUOUS FILAMENT POLYESTER FIBERS——This fabric, particularly in ninon & marquissette constructions, must be heat set before dyeing to obtain dimensional stability and to improve wrinkle resistance. This is necessary because there is considerable residual shrinkage in filament polyester fibers. The usual methods for heat setting can be used. This heat-set fabric may then be satisfactorily dyed on the pressure beam-dyeing machine using recommended disperse dyestuffs at temperatures in the 230-240°F range.

ADVANTAGES OF PRESSURE BEAM DYEING

1) Dyeing at temperatures of 230 to 250°F causes a very great increase in the rate of dye absorption on triacetate and polyester fibers. At these elevated temperatures, there is more complete dye exhaustion, which can result in saving of dyestuff

and chemicals as high as 50%.

2) Further advantage of high-temperature dyeing is the increased rate of penetration of the dye into the individual filaments. Complete penetration usually results in better fastness to washing, crocking and sublimation than with dyeings where the dyestuff has not penetrated well. High-temperature dyeing eliminates the use of carrier or accelerants, which are rather expensive materials, may have toxic effects, may have deleterious effects on the fibers and, if left in the fiber, may seriously effect the lightfastness of the dyeing.

3) Dyeing on pressure beam-dyeing machines allows shorter dyeing times and less labor, thus resulting in lower costs.

4) The beam-dyeing equipment, as made in the United States, is capable of maintaining temperatures of 250°F or even higher. The machine is suitable for lightweight fabrics or fabrics that have a weave open enough to permit liquor flow through many layers of fabric. These machines have been designed for the dyeing of both warp knit fabrics and woven fabrics of synthetic fibers.

While a number of advantages have been listed for the use of the pressure beam-dyeing machine, there are still some disadvantages.

1) Triacetate fabric, and to some extent other synthetic fibers, when dyed medium to heavy shades under pressure on a beam, contain some wild moire. Some of the first batches were very bad. However, as experience has been gained, particularly in regard to batching of the fabric, this defect has become less and less apparent.
2) Some of the dyestuffs normally used for the dyeing of synthetic fibers tend to decompose at high temperatures. This is particularly noticeable in the azo reds; however, there is now available an adequate range of dyes for all shades for all fibers normally dyed on pressure beam-dyeing machines. Most dye manufacturers are aware of this problem and have prepared selected lists for pressure dyeing.
3) Some fibers, particularly wool, when subjected to high temperatures, lose some of their strength. This problem has not been completely resolved, but the fabrics being produced today from polyester/wool blends do have adequate strength.

CONCLUSION

Several years of experience with pressure-dyeing machines has shown this to be a practical approach. Commercial yardages of several types of fabrics have been processed consistently on these machines and it is evident that this type of equipment will continue to be one of the major methods of coloring textiles in the future. A number of mechanical improvements have been introduced to increase control of process.

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Future Papers

The following list is comprised of titles of papers submitted to date for publication in future issues of the Proceedings. Authors and Technical Program Chairmen are requested to examine this list and call our attention to any omissions.

"Fluorescent Brighteners and Bluing Agents in Wool Processing"—Delaware Valley Section

"New Developments in Evaluating 'Wash-and-Wear' Performance" — James R Bercaw, E I du Pont de Nemours & Co, Inc

"Man-made Cellulosic Fibers in 'Wash-and-Wear' "—A B Hilton, G V Lund, and A E Martin Jr. Courtaulds (Alabama) Ltd

"Retrospect and Prospect: Progress and Problems in 'Wash-and-Wear'—Preliminary Report" — Pauline B Mack, Marguerite C Barra, Attelia L Muller, and Elizabeth F Thomas, Texas Woman's Univ

"Recent Advances in the Technology of Dyeing Blends Containing Dacron Polyester Fiber"—Paul L Meunier, E I duPont de Nemours & Co, Inc

"Twelve Million Yards of Natural and Man-made Fibers Dyed on the Swedish Pad-Roll Dyeing Machine" Robert M Lesh, Northern Dyeing Corp

"The Monforts Reactor—A New, Continuous Dyeing machine"—Joachim C Roehl, A Monforts Machine Works

"The I-T Process for Applying Cationic Dyes to Acrylic Fibers"—J A Leddy, Geigy Dyestuffs

"Chemical Principles of Dyeing and Finishing"—Robert A Brooks, E I duPont de Nemours & Co, Inc

"Production of Man-made Fibers" —S L Hayes Jr, E I duPont de Nemours & Co, Inc

"New Developments in Dyeing and Printing Polyester Fibers"—Dorel Marian, Compagnie Francaise des Matieres Colorantes

"New Horizons for Pigment Printing"—Norman S Cassell, Interchemical Corp

"Finishing Blends of Orlon and Cotton in Underwear"—Herbert T Pratt, E I du Pont de Nemours & Co, Inc

"Orlon Cantrece—Du Pont's Newest Sweater Fiber"—Herbert T Pratt, E I duPont de Nemours & Co, Inc

"Laboratory Tested and Approved" —P J Fynn, J C Penney Co

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May 12-13 (Hotel Sheraton-Atlantic, New York, NY); May 12—Council Committees and Research Committees; May 13 (AM)—ECR, TCR; May 13 (PM)—Council

NATIONAL CONVENTIONS
Oct 6-8, 1960 (Sheraton Hotel, Philadelphia, Pa); Sept 28-30, 1961 (Buffalo, NY); 1962 (Southern Region)

DELAWARE VALLEY SECTION
Mar 4 (Beck's-on-the-Boulevard, Philadelphia, Pa); Apr 22 (Ladies Night, Northeast Shrine, Rockledge, Pa); May 20 (Outing, North Hills CC, North Hills, Pa); Sept 16 (Reading, Pa); Nov 4, Dec 9 (Philadelphia, Pa)

HUDSON-MOHAWK SECTION
Mar 27 (Chelsea House, Tribes Hill, NY); May 20 (Ladies Night); June 24 (Annual Outing)

METROPOLITAN SECTION
Feb 19 (Hotel New Yorker, New York, NY); Mar 18, Apr 22 (Kohler's Swiss Chalet, Rochelle Park, NJ); May 20 (Ladies Night—Swiss Chalet); June, 1960 (Outing)

MID WEST SECTION
Feb 6 (Bismarck Hotel, Chicago, Ill; Apr 30 (Schroeder Hotel, Milwaukee, Wis); June 18 (Outing, Cerami's Island View Resort on Browns Lake, Burlington, Wis)

NIAGARA FRONTIER SECTION
Mar 4 (Joint meeting with Ontario Section, CATCC at St Catherines, Ont); Apr 22 (Buffalo, NY)

PACIFIC SOUTHWEST SECTION
Feb 5, Mar 18, May 13 (meetings); July 16 (picnic); Sept 24-25 (outing); Nov 18 (meeting); Dec 10 (dance)

PIEDMONT SECTION
Apr 2-3 (Hotel Robert E Lee, Winston-salem, NC); June 3-4 (Outing—Ocean Forest Hotel, Myrtle Beach, SC); Sept 23 24 (Hotel Charlotte, Charlotte, NC)

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Jan 29 (Johnson's Hummocks); Mar 25 (Providence Engineering Soc); Apr 29 (Student Night); June 24 (Outing—Metacomet Country Club); Sept 23 (Management Night); Oct 28 (Providence Engineering Society); Dec 2 (Annual Meeting—Johnson's Hummocks)

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(continued from page 40)

degraded very rapidly by gamma radiation, readily reacted with acrylonitrile, while Dacron and Orlon, which are degraded very slowly, did not react.

In the case of cotton, with a deposition of 3.5% acrylonitrile, resistance to attack by microorganisms was obtained.

The author discusses the possibility of improving the dyeability of polypropylene fibers by irradiation in adjunct with certain vinyl monomers.

●

The Principal Bleaching Materials and Methods (for cotton)

Campbell, K S, *Textile World* 109, #12, 85, 94-5 (1959); 110, #1, 95, 97 (1960)

Sodium hypochlorite is the principal chlorine bleaching agent used today. It is prepared by flowing chlorine gas into a tank containing caustic soda, soda ash, or a mixture of the two.

The control of pH in hypochlorite bleaching baths is essential to control the rate of bleaching, the whiteness obtained, and the extent of the simultaneous degrading action on the cellulose. Optimum conditions have been empirically established as being in the pH range 9.5 to 11. Temperatures between 55 and 70°F are considered satisfactory.

There are several methods of carrying out the bleaching: 1) piece goods may be saturated with the bleach liquor and then laid down in bins or pits for the bleaching action to take place; 2) the fabric may be piled into a kier and the bleach liquor circulated continuously through the goods; 3) the fabric may be continuously moved through the bleach liquor in a beck or on a jig.

After the bleaching is completed the goods must be washed thoroughly and are usually given an antichlor to remove all residual hypochlorite.

Today peroxide has supplanted chlorine as a bleaching agent to the point where about 80% of cotton is bleached with peroxide, the author states, and about 90% of the cotton that is bleached with peroxide is bleached continuously.

Hydrogen peroxide in stabilized grades containing 35 and 50% H_2O_2 is usually employed. It is necessary at all times to protect the peroxide from sources of contamination, and to keep it from coming in contact with any other types of surfaces than stainless steel, aluminum, porcelain or glass. The bleach bath is corrected to a pH of 10.3 to 10.6 by addition of sodium silicate and/or other alkalis. At temperatures of 180-210°F the time required for bleaching will vary from 45 minutes to five to six hours, depending on the type of goods.

Cotton yarn may also be bleached with peroxide in closed circulating-type machines at a temperature of 240-250°F. The chief advantage is a shortening of the time.

A combination hypochlorite-peroxide bleaching process is also described by the author, who states that this is a common method for bleaching cotton hosiery. The peroxide also serves as an antichlor in this process.

It has been known for many years that chlorine dioxide, ClO_2, will bleach cotton with a minimum of degrading effect. Sodium chlorite produces ClO_2 in acid solution. For practical bleaching the evolution of ClO_2 must be carefully controlled. A pH of 3.5 to 4.0, usually obtained with formic acid, is recommended, and a temperature of 190 to 195°F.

Chlorite bleaching may be carried out continuously or in batch processes. Among the latter the so-called "pad-roll" system is described.

The author states that the chlorite bleach causes less damage to the cotton fiber than either the hypochlorite or the peroxide method. The corrosion and fume problem due to ClO_2 evolution, however, is a serious disadvantage. Various devices for overcoming this trouble are discussed.

●

Flameproofing of Cellulose

Childs, A F, *Dyer* 122, 585-9, Nov 6, 1959

The author briefly reviews the history of flameproofing processes for textiles down to the time of World War II. Developments since then have been along two lines. The first has been the use of organic phosphorus compounds fixed to cotton fabric by means of amide-formaldehyde resins similar to those used in "minimum-iron" finishes. The second has been the attempted reaction of flameproofing substances, both organic and inorganic, with the cellulose molecule itself.

The author comments that further advances in the art of flameproofing will depend on improved understanding of how cellulose burns and how flameproofing agents stop it from burning.

Dry cellulose is not affected by heating below 140°C. Above this temperature it begins to oxidize. Above 300°C it decomposes rapidly to give solid, liquid and gaseous products. The solid residue is mostly carbon, which does not flame but is consumed by after-glowing. The liquid fraction contains water and an involatile tar, together with various organic compounds. The gaseous component consists mainly of CO_2 and CO, with smaller quantities of other materials. The tar is highly inflammable.

Flameproofed cellulose breaks down, when heated, more rapidly than untreated cellulose, giving a greater amount of charred residue and a smaller amount of inflammable liquid and tarry material, and a somewhat greater proportion of water.

The author discusses various theories of flameproofing, and refers particularly to work done in the Southern Regional Research Laboratories and at the Univ of Rhode Island. He states that the requirements for a successful textile treatment are so strict that only a few of the many patents in the field have reached the point of commercial application. He lists the requirements of a good flame-resistant finish as follows: 1) must be flame-resistant; 2) should be glow-proof; 3) permanent to domestic laundering in soap or detergent at the boil; 4) permanent to drycleaning; 5) permanent to commercial laundering; 6) must not alter the dyes on the fabric; 7) must not adversely affect textile properties; 8) must be safe for the worker applying the finish; 9) must not be a skin irritant; 10) if the fabric is chewed, any extract must be nontoxic; 11) it should be possible for the finish to be applied on existing textile finishing equipment; 12) should be as cheap as possible.

The author suggests that the best hope of success seems to be in cheapening the preparation of the phosphorus compounds and improving the methods for applying them to textiles.

Fourteen references to the literature are cited.

●

Du Pont's Latest Spandex Fiber, "Lycra": Prelude to Full Production

Anon, *Man-made Textiles* 36, 40-1, Dec, 1959

"Lycra" spandex* fiber is the result of many years of polymer research and development work. Plans for its commercial production are to be put into effect at once, according to a Du Pont announcement.

This new elastomeric fiber, to which the name "Lycra" has been given, has many outstanding chemical and physical properties which suggest its immediate importance to manufacturers of foundation gar-

*"Spandex" is a generic term describing a manufactured fiber in which the fiber-forming substance is a long-chain synthetic polymer containing at least 85% polyurethane.

ments, swimwear, surgical hose and other elastic products, and an ultimate field of application which may well include furnishings and military and industrial textiles .

The physical properties of Lycra, given in tabular form, include an elongation at break of 650% (comparable with rubber thread), m p 482°F, sp gr 1.00, and moisture regain 0.3% (at 70°F, 65% r h). It will be produced in various deniers from 70 to 560.

In addition to such important characteristics as good strength, uniformity, high abrasion resistance and a long flex life with significant durability, the fiber also has the merit of being white in its natural state (an important feature for foundation garments) and of being readily dyeable in fashionable colors after manufacture into fabric form. The new fiber is also stated to be resistant to ultraviolet light, to weathering, to various chemicals and to cosmetic oils and lotions.

Lycra has affinity for most classes of dyes, and, when desired, it can be dyed to a shade compatible with those of the other fibers in the fabric. The washfastness and lightfastness of dyes on Lycra is generally fair to poor, but by a judicious selection of dyes, a range of shades can be produced with acceptable fastness for most end-uses.

The manufacturers make a number of recommendations for handling Lycra yarn in textile processing. It is pointed out that the yarn as currently produced is not a true monofilament but a multifilament bundle which has been joined together to form the yarn.

It is stated that present finishing procedures for elastic fabrics can be directly applied to fabrics containing Lycra.

•

Produce "Wool" from Man-made Fibers

Anon, *Can Textile J 76*, 71, Nov 13, 1959

Russian scientists claim to have developed a synthetic wool ten times stronger and ten times cheaper than the natural product. It will go into production soon, according to a recent Russian report, a translation of which has been issued by the U S Dept of Commerce, Washington, DC.

Basic ingredients are lignin and cellulose, both constituents of wood. Molten lignin is mixed with epichlorohydrin, derived from cellulose by a complicated chemical process. "Fibers of very high strength are produced by spinning from the molten mixture," says the report. "These are highly resistant to the effects of moisture and can be dyed with facility."

"According to tentative calculations, fibers derived from glucose oil lignin obtained from wood wastes will cost no more than 9,000 rubles (about $900 at the tourist rate of exchange) a ton, while the cost of pure wool is 90,000 rubles. It must also be taken into consideration that the synthetic fiber will have a tensile strength *ten times* higher than that of wool."

The report also tells of new methods for obtaining glucose, or "corn sugar", from wood at a cost 40% less than present methods, and using it as a major material in the synthesis of a large variety of plastics.

(Further details of the new fiber will be awaited with interest and possibly some skepticism—*Ed.*)

• PATENT DIGEST •

PAUL WENGRAF

DYEING OF SYNTHETIC FIBERS———Emulsified Aromatic Esters as Assistants

C,4,07

U S Pat 2,881,045 (Am Cyanamid—Mecco et al—April 7, 1959)

Various carriers, such as phenylphenols, monochlorobenzene, etc, have been described in the past as suitable carriers for use in the dyeing of hydrophobic synthetic fibers. The current specification enumerates (in column 1, lines 47-58) some of the major defects inherent in the use of these products.

In contrast, emulsions of esters of aromatic monocarboxylic acids were found to be suitable dyeing assistants, which permit level dyeing of synthetic fibers in deep shades. Esters useful for this purpose are methylsalicylate, methylbenzoate, and many others (column 2, lines 54-67). The emulsifying agents may be of the anionic, nonionic or cationic type or mixtures of anionic and nonionic surfactants. A critical factor, however, is the ratio of emulsifier to ester. Too much emulsifying agent causes poor fixation (thus weaker shades) and must be avoided. The upper limit is given as one part emulsifier to two parts ester, but satisfactory results have been observed with one part emulsifier to 5-50 parts ester. The presence of emulsifiers was found to be absolutely necessary: no improved results could be attained using non-emulsified esters. The dyeing assistant must be emulsified with a small amount of aqueous phase before it is added to the dyebath; the emulsification process cannot be carried out in the dyebath itself.

Improved dyeing effects reportedly are not limited to any special group of dyes. The method may be used with acetate, vat, acid, etc, dyes and pigments; for dyeing acrylic fibers by the cuprous-ion process; and also for applying optical bleaching agents, such as coumarine derivatives.

Example: A Dacron fabric was immersed in water, and an aqueous emulsion of 400 parts methylsalicylate, in which 20 parts of a polyethoxylated p-octyl phenol plus 20 parts of dioctyl sodium sulfosuccinate had been dissolved, was stirred in. The cloth was worked in this bath at 100°F until uniform penetration occurred. Then the scarlet dye CI Disperse Red 1 was pasted with water and added. Following a dyeing at the boil, a deep scarlet color reportedly was obtained; an identical fabric dyed with methylsalicylate in the absence of an emulsifier appeared dull and spotty.

Among the references cited by the Patent Office:

Ger Pat 741,457 (I G Farben /1944): Polyvinyl fibers may be dyed with dispersions of water-insoluble dyes plus dispersed water-insoluble ketones, carboxylic, carbamic or phosphoric acid esters; or derivatives thereof.

U S Pat 2,362,376 (Am Viscose /1944) refers to the dyeing of vinyl polymer fibers in the presence of various aryl- or alkylesters, eg, dibenzylphthalate or glycerol dibenzoate.

U S Pat 2,736,631 (Celanese Corp/1956: di(diethyl-aminoethyl-) phthalate is incorporated into dyed cellulose acetate fabrics to prevent acid fading. [Cf *Am Dyestuff Reptr* 45, 957 (1956)].

News of the Trade

Rockefeller Elected NAFTF President

Godfrey S Rockefeller was elected president of the National Association of Finishers of Textile Fabrics at its 46th annual meeting held last month in New York. Mr Rockefeller, president of Cranston Print Works Co, Cranston, RI, succeeds Lawrence Marx Jr, United Merchants & Manufacturers, Inc, who was elected chairman of the Executive Committee.

Richard D Wood Jr, Millville Mfg Co, Millville, NJ, and Ernest J Chornyei, Bradford Dyeing Association (USA), Westerly, RI, were elected vice presidents of the Association. Joseph E Hoesl, NAFTF secretary, was reelected treasurer.

In his annual report to the membership, the secretary reviewed the activities of the Association and in a summary of the economic conditions of the industry he stated:

"Cotton finishing plants made favorable gains during 1959. The production level averaged an estimated six percent above 1958, but it is still about five percent below the peak reached in 1955. Estimates at the year end indicate that total cotton goods finished amounted to 7.7 billion yards as compared with 7.2 billion last year. Of this total, bleached goods accounted for 3.3 billion yards—up five percent; plain dyed goods increased over 11 percent to 2.6 billion yards; whereas the output of printed goods of 1.7 billion yards was only about two percent above last year.

"The current upturn in finished cotton goods production began in the 4th quarter of 1958 and in each successive quarter of 1959 it showed a gain over the same quarter of the previous year. At the year end, it is estimated that bleached goods production was only four percent under the peak production reached in 1956 for this class of finishing. Dyed goods production was less than two percent under its peak year of 1955. However, printed goods output is about 18 percent below the peak it hit in 1955.

"An interesting observation about the current upturn in cotton finished goods output is its similarity to conditions in 1954 when production also started upwards in the 4th quarter of the year. In the following eight quarters (two years) the production level gained steadily until the 4th quarter of 1956. It then reversed itself and went downwards for a similar eight-quarter period to the latter part of 1958 when the current upturn began. A simple assumption would be that the current upturn of five quarters will continue for another three quarters in 1960 to complete the cycle.

"What happens to the production level in 1960 depends on several economic and political factors: among these are the need for a continuing high level of general business activity; avoiding buildup of surplus inventories through careful control by management of textile output to consumer demand, and, finally, concerted effort on the part of the textile industry to convince the administration of the vital need to 'put a lid' on the mounting tide of imported fabrics and apparel."

Also elected at the annual meeting to serve as members of the Executive Committee were the following: Robert Amory, Springs Cotton Mills, Grace Bleachery, Grace, SC; Robert Bendheim, Rock Hill Prtg & Fin Co, Rock Hill, SC; Elliot Broadbent, Sayles Finishing Plants, Saylesville, RI; Homer Carter, Pepperell Mfg Co, Pepperell, Ala; J Gordon Coles, Fairforest Fin Div, Reeves Bros, Spartanburg, SC; Sydney M Cone Jr, Cone Mills Corp, Greensboro, NC; E R Farrell, Mansfield Bleachery, Mansfield, Mass; A E Gutman, Harodite Fin Co, North Dighton, Mass; R H Jewell, Crystal Springs Bleachery, Chickamauga, Ga; John McConchie, The Kendall Co, Bethune, SC; Royce N Pharr, USF-Aspinook Fin Div, Norwich, Conn; W F Robertson, Riegel Textile Corp, Ware Shoals, SC; Robert D Sellers, Southern Bly & Prt Wks, Taylor, SC.

In addition, the following members continue to serve unexpired terms on the Executive Committee: J Marshall Cole, Cold Spring Bleachery, Yardley, Pa; W R McIntyre, Jos Bancroft & Sons Co, Wilmington, Del; James Harrell, Delta Fin Div, J P Stevens & Co, Wallace, SC; Arthur G Poor, Standard Bly & Prtg Co, Carlton Hill, NJ; Julian Robertson, North Carolina Fin Co, Salisbury, NC.

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Althouse Joins TRI

John F Dillon, president, Textile Research Institute, Princeton, NJ, announced last month that the Althouse Chemical Co, Reading, Pa, has joined the Institute as an active member.

Fiber Society Announces Program for Spring Meeting in New Orleans

The program for the spring meeting of the Fiber Society, to be held at the Roosevelt Hotel in New Orleans on March 10 and 11, has been announced by Society President Ernest R Kaswell, Fabric Research Laboratories, Inc, Dedham, Mass.

Chairman of the opening session will be Burt Johnson, National Cotton Council. He will introduce James R Meyer, U S Department of Agriculture, Agricultural Experiment Station, Stoneville, Miss, who will discuss "The Use of Species to Modify and Improve the Lint of Upland Cotton." Also during the first session Carl M Conrad and David J Stanonis will discuss "Fine Structure and Mechanical Properties of Highly Cyanoethylated Cotton Yarns."

The second session will be chaired by Milton M Platt, Fabric Research Laboratories, Inc, and will include the following discussions: "Relationship Between Fiber Properties and Fabric Wrinkle Recovery," W W Daniels, E I du Pont de Nemours and Co, Kinston, NC; and "The Bending Behavior of Single Fibers and Wrinkle Resistance," John B Miles, The Chemstrand Corp, Decatur, Ala.

The morning session of Friday, March 11, will have as its chairman E V Painter, Johnson & Johnson, Chicago, Ill. Technical presentations will be: "Adhesion Mechanism Between Fibrous Materials and Resins in Composite Laminates," G A Stein, Archer-Daniels-Midland Co, Minneapolis, Minn; and "The Resistance of Fibrous Mats to Fluid Flow," W L Ingmanson, Institute of Paper Chemistry, Appleton, Wis.

Fiber Society Vice President Stanley Backer, Massachusetts Institute of Technology, Cambridge, Mass, will be chairman of the final session, which will include: "The Differential Thermal Analysis of Textile Materials," Robert F Schwenker Jr and Louis R Beck Jr, Textile Research Institute, Princeton, NJ; and "Comfort of Textiles in Relation to Simultaneous Transfer of Heat and Moisture," Lyman Fourt, Harris Research Laboratories, Washington, DC.

Further information on the meeting and program may be obtained by writing to Fiber Society, PO Box 405, Athens, Ga.

"Man-made Textile Encyclopedia" Published

Textile Book Publishers, New York, announced on December 21 the publication of "Man-Made Textile Encyclopedia," described as "the first fully definitive account of the workings of the entire man-made fiber and textile industries." The Encyclopedia is designed to serve every interest in the textile industry, from chemical raw materials to garment manufacture, renovation, marketing, and economics. J J Press, chief of the Textile Branch of the U S Navy Clothing and Textile Research Laboratory and editor-in-chief of the Encyclopedia, has worked in close association with an international group of 149 textile experts who have contributed comprehensive and completely up-to-date articles to the work. An indication of the enormous amount of information contained in the Encyclopedia—and of the efforts of the editor, contributing authors, and the publisher in its preparation and production—is given by the size of the book. It contains 944 pages, 8¼ × 11¼ in a two-column format, and is illustrated throughout with 561 half-tone or line illustrations, and 370 charts and tables.

A special prepublication price was offered until publication date; The established list price now in effect is $27.50. Orders for the "Man-Made Textile Encyclopedia" should be directed to Textile Book Publishers, Inc, 250 Fifth Avenue, New York 1, N.Y.

Textile Book Publishers is a division of Interscience Publishers.

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SunOlin Facilities to be Expanded to Manufacture Ethylene

Plans to expand facilities at SunOlin Chemical Co in North Claymont, Del, to include the manufacture of ethylene were announced recently by James I Harper, president of SunOlin. The new plant will cost approximately $15,000,000.

Ultimate capacity will be nearly 200,000,000 pounds a year, Mr Harper said. A portion of the ethylene will be converted to ethylene oxide and glycol to meet developing needs on the East Coast. Further details will be announced later.

SunOlin is an equally owned affiliate of Sun Oil and Olin Mathieson Chemical Corp.

Dry gas from Sun Oil's Marcus Hook, Pa, refinery will be used for the production of ethylene. The SunOlin plant in Delaware is adjacent to Sun's refinery.

Details of the transaction whereby Celanese Corporation of America acquired from B F Goodrich Co world rights to make and market Darvan were announced last month by Harold Blancke, president of Celanese (left) and J W Keener, president of B F Goodrich, shown examining a sweater knitted with Darvan yarns.

Celanese Acquires Rights to Darvan

World rights to make and market Darvan nytril fiber have been purchased by Celanese Corp of America from The B F Goodrich Co. The acquisition will substantially expand markets for Celanese chemical products and further diversify the company's line of contemporary fibers, it has been stated.

Darvan has significant potentialities in a variety of apparel end uses, both in woven and knitted fabrics. Under the transaction, Celanese acquires the patents, trademark, technical know-how and world rights to produce and market the fiber. B F Goodrich retains rights outside the textile fibers field, including plastics applications.

Darvan can be used 100% or in blends with wool, cotton or with other man-made fibers. It is said to contribute a soft luxurious feel and appearance, high bulk, good shape retention, serviceability in wear and good washability.

At present, Darvan is being produced in staple form in a plant at the B F Goodrich Chemical Co's Development Center in Avon Lake, Ohio. B F Goodrich will operate the plant for Celanese until such time as Celanese starts producing the fiber with its own facilities.

Along with helping broaden Celanese fiber markets, Darvan has a logical place with Celanese from a raw materials standpoint. Acetic acid and vinyl acetate, representing the source of nearly three-fourths of Darvan's chemical composition, both are produced by Celanese Chemical Co at its Pampa, Tex, plant.

Patent Office Sponsoring Chemicals and Synthetics Exhibit

The U S Department of Commerce has selected 20 leading chemical manufacturers to participate in a Chemicals and Synthetics Exhibit to be sponsored by the Patent Office in Washington, DC, from February 15 through March 11.

The exhibit will be held in the lobby of the Department of Commerce and will be open to the public. According to the Commissioner of Patents, the purpose of the exhibit is "to make the beneficial aspect of our free-enterprise system better understood." It is estimated that more than 75,000 people will view the program during its month-long display.

Arnold, Hoffman & Co, Inc, one of the participating concerns, plans to feature representative products from its line of dyes and chemical auxiliaries and to show how these products benefit the consumer. A variety of fabrics will be used to illustrate how modern dyes and finishes, such as those that Arnold, Hoffman produces, contribute to the beauty and durability of modern textiles.

To emphasize the wide range of uses for dyes and finishes, the Ahco display will show models dressed in ensembles of both all-natural fabrics and all-synthetic fabrics. The benefits afforded by the U S patent office in the development of Ahco products will be the underlying theme of the display.

•

TDI Assumes Direction of Two Industry Service Bureaus

Direction of two textile industry service bureaus, namely the Design Registration and Trademark Bureaus, was transferred on January 1 from the American Cotton Manufacturers Institute Inc to the Textile Distributors Institute, Inc.

TDI has taken over the two bureaus as part of its expanded program and as a service to the industry. The services will be available to both members and nonmembers of TDI.

The bureaus, which were under the auspices of the National Federation of Textiles before its consolidation with ACMI about two years ago, will continue temporarily in the ACMI office at 10 East 40th St, New York, until they can be located at the present premises of TDI at 469 Seventh Ave. Present personnel will be retained to operate the bureaus under the direction of Hilda Wiedenfeld, TDI executive director.

Softness sold this towel...

and CARBIDE's amines gave it that softness

Textile softeners, based on CARBIDE's amines, give linens and cottons the softness that makes them sell. And, with the complete selection of amines available from Union Carbide, you can make textile softeners that give the right kind of softness for best sales appeal in finished goods. Cationic mill-softeners, prepared by fatty acid esterification or amidification of CARBIDE's amines, offer a full range of softening effects. Here are seven amines for your evaluation:

- Triethanolamine
- Diethanolamine
- N-Aminoethyl Ethanolamine
- N-Methyl Diethanolamine
- Diethylene Triamine
- Dimethylamino Propylamine
- Diethylamino Propylamine

Remember, whether you need amines . . . UCON textile lubricants . . . anti-foaming agents for scouring and mercerizing . . . water-insoluble solvents to achieve deeper and fuller shades in dyeing . . . monomers for fiber syntheses . . . ethanolamines and TERGITOL nonionic surfactants for detergents . . . or other organic chemicals for finishing agents and lubricants . . . you'll find them at CARBIDE. With more than one hundred CARBIDE textile chemicals to choose from, you can take advantage of the increased savings of bulk buying in mixed tank car and mixed tank truck lots, or mixed carload lots in drums.

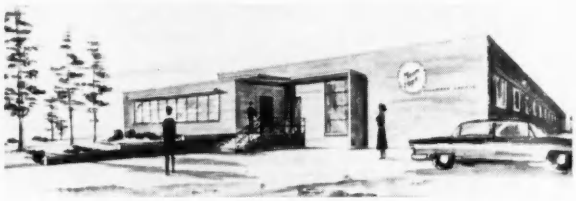
For information and help from experienced Technical Representatives, call your nearest CARBIDE sales office or write to: Union Carbide Chemicals Company, Room 328, Department J, 30 East 42nd Street, New York 17, N. Y.

UNION CARBIDE CHEMICALS COMPANY

UNION CARBIDE

The terms "Tergitol", "Ucon" and "Union Carbide" are registered trade marks of UCC.

Artist's rendering of the new distributing center which is being built in Lyndhurst, NJ, by Becco Chemical Division.



Artist's conception of Mount Hope Machinery Company's new plant at Charlotte, NC.

Becco Starts Construction of New Distributing Center in NJ

Construction was began last month in Lyndhurst, NJ, on a new distributing center for the Becco Chemical Division of Food Machinery and Chemical Corp, whose headquarters and principal production plant are in Buffalo, NY.

The new distributing center, which is expected to be completed by May 1, will replace Becco's present center in Jersey City, which was established in 1929. It reportedly will provide larger and more efficient storage and distribution facilities for the company's products. It will also be more conveniently situated with respect to rail and road transportation facilities than the Jersey City center, and thus enable Becco to provide faster deliveries to its customers in the New York-New Jersey Metropolitan area as well as to those elsewhere in the Middle Atlantic states.

The new Lyndhurst center will receive bulk shipments of hydrogen peroxide and other peroxygen chemicals from Becco's main production plant in Buffalo, and will distribute these in smaller quantities to customers in the Metropolitan area.

Becco maintains similar distributing centers in Framingham, Mass, and Charlotte, NC. Customers in the Midwest and Far West are served directly from Buffalo or from Becco's other production plant in Vancouver, Wash.

The new Lyndhurst distributing center occupies part of the former site of the Lackawanna Railroad's Kingsland car and locomotive maintenance shops. The Becco property is a triangular plot fronting on Schuyler Avenue, and totaling approximately 75,000 sq ft. On this plot, Becco will construct a warehouse containing approximately 22,000 sq ft of storage space and an adjoining office block providing about 2,500 sq ft of space. A railroad siding leading into the property will provide convenient unloading facilities for the tank cars arriving from the production plant in Buffalo.

Matthew A Noonan, Becco's district manager in New York, will direct the distributing operations of the new center. Anthony Wieand, distribution supervisor at Becco's present Jersey City center, will continue to be in charge of the new Lyndhurst center, which will employ about five men.

•

Carwin Absorbs Subsidiary

The Carwin Company absorbed its wholly owned subsidiary, Carwin Polymer Products, Inc, on December 31, 1959. The business of developing, manufacturing, and marketing products previously carried on by the subsidiary will now be conducted by The Carwin Company, Polymer Products Division, whose personnel remains unchanged.

•

Rodney Hunt Becomes Exclusive Agent for Bentley Rolls

The Industrial Roll Division of the Rodney Hunt Machine Co, Orange, Mass, has announced it appointment as exclusive United States agents for David Bentley, Ltd, Manchester, England. David Bentley, Ltd is reported to be the oldest manufacturer in the world of all kinds of filled calender rolls used for friction calendering, schreiner calendering and embossing.

This arrangement makes available in the United States Bentley's unique wood-fiber roll for water and starch mangling. It is expected that this particular roll will replace many solid wood and rubber-covered rolls because of its alledgedly superior properties in the high extraction of moisture.

The wood-fiber roll will be marketed in Canada by the Rodney Hunt Machine Co, through its Canadian agents, W J Westaway Co, Ltd, with offices in Montreal, Que, and Hamilton, Ont.

A comprehensive 36-page brochure on Bentley Calender Rolls, as well as an 8-page brochure on Bentley Wood-Fiber Rolls, may be obtained without obligation by writing to Industrial Rolls, Div, Rodney Hunt Machine Co, Orange, Mass.

Construction Begun on Mt Hope Machinery Plant in NC

Mount Hope Machinery Co, Taunton, Mass, manufacturers of expanders and other equipment used in the manufacturing and processing of textiles, has announced that construction has already been started on a new and modern plant in Charlotte, NC. It is expected that the company's Southern organization will move from its present Charlotte location to its new plant on Ross Drive in the Spring.

The new plant reportedly will have complete facilities for the manufacture and servicing of all Mount Hope products. It will also house the Mount Hope Southern Sales Office, under the direction of Slaughter Machinery Co of Charlotte.

•

34 Industries Represented at NTTCIW Meeting

Scientific, technical and mangement representatives of 34 major industries met at the Conrad Hilton Hotel in Chicago, December 15-16 to discuss the year's major developments to reduce or control the amount of industrial waste flowing into the rivers, lakes, and streams of the United States.

This was the ninth annual meeting of the National Technical Task Committee on Industrial Wastes, composed of 65 scientists and engineers. The United States Public Health Service also was represented.

The Committee was established in 1959 at the invitation of the Surgeon General of the United States. Its function is to cooperate with the Public Health Service in carrying out the purposes of the Federal Water Pollution Control Act to safeguard the public health and welfare by the prevention and control of water pollution.

Representing the Textile Industry were Percival Theel, coordinator of research, Philadelphia Textile Institute, Philadelphia, Pa, and R Hobart Souther, Cone Mills Corp, Greensboro, NC.

One in a series of interviews with the men who are "Virginia"

"SO_2 as a neutralizer is very easy to handle. Fabrics leave the washer completely free of chlorine and alkali . . . and will not tender at the dry cans"

The efficiency of liquid sulfur dioxide as a neutralizer and the savings it offers in time and labor are discussed by James Todd, a technical representative for Virginia Smelting Company.

James Todd, "Virginia" technical representative

Q Is SO_2 the most efficient antichlor available for reducing and neutralizing?

A Yes. We have been working in close coordination with the textile industry for many years and we have yet to find an antichlor that offers as many over-all advantages as SO_2.

Q Do all bleachhouses use SO_2 for this purpose?

A No, I regret to say they don't. Some use SO_2 salts, some acetic acid, and perhaps a few still use sulfuric or hydrochloric acid. But I believe if every bleachhouse knew the advantages SO_2 offers, they would all be using it right now.

Q Is "Virginia" liquid sulfur dioxide especially well suited for this application?

A Definitely. Our SO_2 is shipped in steel cylinders that are easy to handle and install where needed. And its purity —99.98%—guarantees the bleachhouse operator that he is adding no impurities that can stain the fabrics or leave residues that must be disposed of later.

Q Why is liquid SO_2 more practical than SO_2 salts or the acids you mentioned?

A Salts can build up in the sour box and slow down the speed and efficiency of neutralization. Sulfuric and hydrochloric acids present special problems. Sulfuric acid is extremely difficult to remove from fabrics and requires unusually large volumes of water and a lot of washing. Otherwise, when the goods go to the dry cans, heat and evaporation will concentrate the sulfuric acid and any hypochlorites left on the fabric. This tenders cotton and affects cellulose compounds, too. Hydrochloric acid also has to be handled with great care, as its strength is 20 times that required of SO_2. And all the acids are difficult to handle, compared with SO_2.

Q Is SO_2 easy to use?

A Extremely easy. To begin with, you don't have the worry of fragile glass carboys or big drums that the operator has to pour from—a problem with acetic acid, H_2SO_4 or HCl. With SO_2 we provide a simple control apparatus on the steel cylinder that permits the operator to diffuse the SO_2 directly into the water. It's very easy to control the quantity—to add just the right amount. It never varies in purity, never decomposes in the container, and it goes a long way in the bleachhouse.

Q How does it compare in cost with other neutralizers?

A Well, some of the others are a little cheaper, either initially or because their high strength means that less is used at a time. But, on the other hand, it takes, for example, 2.02 lb. of 66° sulfuric acid to neutralize 1 lb. of hypochlorite and 1 lb. of alkali, whereas less than 1 lb. of SO_2 will handle a pound of hypochlorite and one of alkali. And an SO_2 antichlor bath can be used for several days without the buildup of undesirable salts that occurs with many other neutralizers. Also SO_2 is not only easy to use, but very safe. And there is no risk of tendering fabrics. In the long run, a bleachhouse can save substantially by using SO_2 and improve product quality at the same time.

Q Why doesn't SO_2 tender the fabrics?

A Because any SO_2 left on the fabric goes off into the air at the dry cans. In solution, of course, it is sulfurous acid. But the heat at the cans breaks it down quickly and the water goes off in steam, the SO_2 in a faint vapor. I might also mention that since SO_2 is an absolutely certain way of killing off all chlorine, many bleachhouses have been able to increase the strength of their chemic and get a better white.

FOR MORE INFORMATION ON SO_2 USES IN THE TEXTILE INDUSTRY

just write us. We will send technical information—or have a "Virginia" man call on you if you wish.

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• NAMES IN THE NEWS •

Ainsworth

Dougherty

Doyle

Lavoie

Bruce Ainsworth has become president of the Harchem Division, Wallace & Tiernan Inc. In this capacity he will be responsible for all Division activities including research, product development, production and sales.

Mr Ainsworth was formerly assistant to *Charles H Rybolt*, vice president in charge of W & T's Chemical Divisions.

•

Werner von Bergen, associate director of research for J P Stevens & Co, has been chosen as the 1960 recipient of the Harold DeWitt Smith Memorial Medal given by Committee D-13 of the American Society for Testing Materials.

Mr von Bergen will be the 11th recipient of the annual award, which will be made on Thursday, March 3, at the Sheraton-Atlantic Hotel in New York, NY.

The medal, donated by the Fabric Research Laboratories, Inc, is a testimonial to the memory of Harold DeWitt Smith, who pioneered in the engineering approach to evaluation and utilization of textile fiber properties.

•

Philip M Dinkins, president and chief executive officer of General Aniline & Film Corp, has been awarded the Junior Achievement Bronze Medal for outstanding service to youth and the community by the Junior Achievement of Union County, Inc, New Jersey.

General Aniline & Film Corp has sponsored a Junior Achievement "company" for the past 10 years as well as contributing to the development and growth of the Junior Achievement program.

•

Norman Gordon has been appointed to the sales staff of the Onyx Oil & Chemical Co, Jersey City, NJ.

Mr Gordon will be assigned to the Chicago office and will call on accounts in Missouri, Kentucky, Kansas, Arkansas and Oklahoma.

Mr Gordon was formerly with Polak & Schwartz.

L Paul Dougherty and *Jack Doyle* have been added to the Organic Chemical sales staff of Emery Industries, Inc. Mr Dougherty will be assigned to the New England sales territory, while Mr Doyle will cover a territory that includes West Virginia, southeastern Ohio, western Pennsylvania, and western New York State.

Mr Dougherty succeeds *Walter R Paris*, who resigned earlier this year. Prior to joining Emery, he was associated with Hagan Chemicals & Controls, Inc, as a chemical sales representative. He will make his headquarters in Emery's Boston office.

Mr Doyle has previously been associated with the Bowman Steel Co.

•

Nissl

John G Nissl, New York sales manager for Carbic-Hoechst Corp, retired January 1, after 38 years of service. Mr Nissl, before joining Carbic, was employed by Griesheim-Electron in Germany.

A testimonial dinner for Mr Nissl was held on December 22, 1959 at Luchow's Restaurant in New York for thirty friends and associates. Mr and Mrs Nissl are taking a three-month's trip before returning to their home in Huntington, NY.

•

Robert A Bondurant Jr has been appointed manager of the Chlor-Alkali Division of Food Machinery and Chemical Corporation. Before his appointment, Mr Bondurant was resident manager of the division's soda ash facility near Green River, Wyo.

E P Lavoie has joined Wica Chemicals, Inc, as general sales representative. This move marks Mr Lavoie's 25th year in service to the textile industry.

Mr Lavoie is active as a Sectional Committeeman in the Piedmont Section, AATCC.

•

Henry Millson of American Cyanamid Company addressed ninety-five members and guests of the Ontario Section of the Canadian Association of Textile Colourists & Chemists at the Ontario Agricultural College, Guelph, Ontario, in December.

The regular members were joined by members of the Chemical Institute of Canada and students of the Textile Section of the Hamilton Institute of Technology who had been the guests of Artex Woolens Ltd, Hespeler, Ont, earlier in the day.

Dr Millson's lecture, followed by slides and films covering "Microscopical Studies of Dyeing" and "Time Lapse Motion Picture Photography", was the forerunner of an extensive question period.

•

Robert W Cairns, director of research for Hercules Powder Co, has been elected a member of the board of directors. Dr Cairns has been director of research since 1955. He began his career with Hercules in 1934 as a research chemist.

•

Robert Strain has been appointed industry manager for polymer and surfactant chemicals by the Armour Industrial Chemical Co, Chicago, Ill.

Mr Strain joined Armour as a salesman in 1950 and has been headquartered at Indianapolis since that time.

•

United States Testing Company, Inc, has announced the appointment of *Howard L Averbach* to the staff of the paper laboratory and *John Twarog* to the staff of the Dye Laboratory of the Textile Division.

New uses are still being found for

GROCO RED OILS & WHITE OLEINES

SPECIFICATION	WHITE OLEINES		RED OILS			
	GROCO 6 USP	GROCO 5L Low Linoleic	GROCO 2	GROCO 4	GROCO 8	GROCO 18
Titre	2° – 5°C.	2° – 5°C.	3°C. max.	4° – 6°C.	8° – 10°C.	18° – 20°C.
Titre	36° – 41°F.	36° – 41°F.	37.4°F. max.	39.2°–42.8°F.	46.4°–50.0°F.	64.4° – 68°F.
Color 5¼"* Lovibond Red	1 max.	1 max.	1 max.	1 max.	1 max.	2 max.
Color 5¼"* Lovibond Yellow	8 max.	8 max.	10 max.	10 max.	10 max.	15 max.
Color Gardner 1933	2 max.	2 max.	—	—	—	—
Unsaponifiable	1.0% max.	1.0% max.	1.5% max.	1.5% max.	1.5% max.	1.5% max.
Saponification Value	199 – 204	201 – 206	198 – 203	198 – 203	198 – 203	204 max.
Acid Value	198 – 203	200 – 205	197 – 202	197 – 202	197 – 202	203 max.
% F.F.A. as Oleic Acid	99.5 min.	99.5 min.	99 min.	99 min.	99 min.	99 min.
Iodine Value (WIJS)	95 max.	90 max.	95 max.	94 max.	92 max.	85 max.
Refractive Index 50°C. (Average)	1.4500	1.4490	1.4505	1.4500	1.4495	1.4485
Total Polyunsaturated Fatty Acids	—	3.5% max.	—	—	—	—
COMPONENT FATTY ACIDS						
C14 Myristic	5%	6%	4%	4%	5%	5%
C16 Palmitic	3%	2%	3%	5%	6%	10%
C18 Stearic	1%	—	1%	1%	2%	4%
C18 Oleic	82%	89%	82%	80%	77%	72%
C18 Linoleic	8%	2.5%	9%	9%	9%	8%
C18 Linolenic	1%	0.5%	1%	1%	1%	1%
MACKEY TEST: Time to reach 105°C.	over 7 hrs.	over 7 hrs.	over 4 hrs.	over 4 hrs.	over 4 hrs.	over 3 hrs.

For more information on A. Gross fatty acids, send for new edition of "Fatty Acids in Modern Industry."

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The appointment of committee chairmen of the Synthetic Organic Chemical Manufacturers Association for 1960 has been announced by *Samuel G Baker*, president of the Association, and chairman of its Information Policy Committee.

K E Mulford, Atlas Powder Co, was appointed chairman of the Additives Committee, with *E I Stearns*, American Cyanamid Co serving as chairman of the Dyes Subcommittee of that committee. *P R Rector*, Union Carbide Corp, will head the Government Reports Committee; *F E Bennett*, Publicker Industries, Inc, the Membership Committee, and *E M May*, Otto B May, Inc, the Research Committee.

The International Commercial Relations Committee, which functions through the operations of eleven subcommittees, will be headed by *C K Black*, E I duPont de Nemours & Co, Inc. Dr Black named his subcommittee chairmen as follows: *K R J Horner*, Ciba Co, Inc, for the Common Market-Free Trade Areas Subcommittee; *C W French Jr*, Pfister Chemical Works, for the Customs Subcommittee; *A C McAuliffe*, American Cyanamid Co, for Defense Subcommittee; *J M Fasoli*, American Cyanamid Co, for the Escape Clause-Antidumping Subcommittee; *W D Hoag*, Allied Chemical Corp, for the Exports Subcommittee; *L E Lloyd*, Dow Chemical Co, for the GATT Subcommittee; *D MacArthur*, Koppers Co, Inc, for the International Cooperation Administration Subcommittee; *H F Shattuck*, Monsanto Chemical Co, for the Revision of Tariff Act Schedules Subcommittee; *Dr Black*, for the Tariff Legislation Subcommittee; *M A Dahlen*, E I duPont de Nemours & Co, Inc, for the Technical Advisory Subcommittee; and *N C Babcock*, Union Carbide Corp, for the Trade Agreement Negotiations-Peril Point Subcommittee.

Mr Baker appointed the following as chairmen of internal committees: Mr Babcock, for the Budget and Assessment Committee and the Pension Committee; *R W Daniels*, American Cyanamid Co, for the Outing Committee; *P L Filter*, Dow Chemical Co, for the Programs Committee, and *C A Gernstacker*, Dow Chemical Co, for the Steering Committee.

OBITUARY

REES F TENER, assistant chief of the Testing and Specifications Section, National Bureau of Standards, Washington, DC, died suddenly on December 25, 1959.

He joined the staff of the National Bureau of Standards in November 1924 and served in the Rubber Section until July 1930, then in the Testing and Specifications Section until his death.

Mr Tener received the Department of Commerce Meritorious Service Award in 1953 for his outstanding work in the development of Federal specifications and test methods. He was particularly active in the work of the American Society for Testing Materials and served as chairman of subcommittees of Committees D-11 on Rubber and Rubber-Like Materials and D-13 on Textile Materials. The National Bureau of Standards was represented by Mr Tener on the American Standards Association's L-14 on Textile Test Methods and L-24 on Institutional Textiles.

• NEW PRODUCTS AND DEVELOPMENTS •

Procion Brilliant Purple H3RS

Procion Brilliant Purple H3RS, recently introduced by Arnold, Hoffman & Co, Inc, is a homogeneous reddish purple fiber-reactive dye said to possess excellent washfastness and excellent lightfastness properties.

Of special interest to dyers and printers of cotton and viscose rayon, Procion Brilliant Purple H3RS reportedly builds up well to deep shades. It is an "H" type Procion dye, and therefore requires steaming for fixation after printing, but its lower reactivity is said to contribute to its very good print paste stability.

In dyeing operations, Procion Brilliant Purple H3RS reportedly exhibits very good wash, light and perspiration fastness on cotton and viscose rayon, and good fastness to hypochlorite on cotton. It is said to be highly soluble and therefore particularly suitable for padding applications, but it can be applied also by any of the methods generally recommended for Procion "H" brand dyes. It is stated that, in batch dyeing techniques Procion Brilliant Purple H3RS shows good color value and good building up properties on viscose rayon, and moderate affinity for cotton.

Procion Brilliant Purple H3RS is not dischargeable.

With the introduction of this dyestuff, the total range of Procion fiber-reactive dyes now numbers twenty-seven. Developed by Imperial Chemical Industries Ltd of England, they are manufactured in the USA by Arnold, Hoffman.

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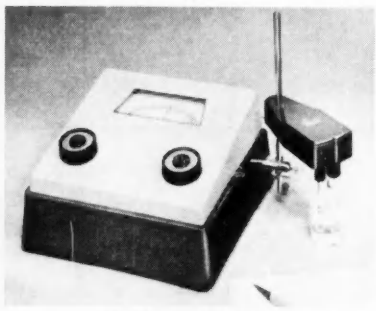

Metrion pH Meter

Metrion pH Meter

The Metrion pH meter, announced last month by Coleman Instruments, Inc, Maywood, Ill, is said to deliver precision usually found only in high-priced pH meters. It also is said to offer an extended range, utilizing large, clear, duplex scales covering 0 to 10.0 pH and 4.0 to 14.0 pH. It is stated that the Metrion uses Coleman screw-base electrodes and also accepts all other modern pH electrodes. According to the manufacturer, this unusually convenient electrode-mounting system adapts perfectly to titrations and a wide range of sample volumes.

The Metrion is housed in a case with high resistance to shock, acids and stains. It is priced at $139.00.

Write for Bulletin B-257, Coleman Instruments, 42 Madison Street, Maywood, Ill.

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Atco SA

Metro-Atlantic, Inc has announced the addition of Atco SA to its line of finishing auxiliaries. Atco SA is a newly developed product designed to produce antislip properties on synthetic fabrics where seam slippage is a problem. An outstanding advantage of this product is said to be that it can be used on the acid side with other finishing materials and dyefixing agents. It also is said to possess the property of building hand, particularly when applied in the finish bath. Metro-Atlantic advises that Atco SA is used most satisfactorily with finishes that can be maintained slightly on the acid side.

Eccoblanc W-45-B

Eccoblanc W-45-B—a new pigment white for roller printing or discharge printing—is termed the "whitest white obtainable" by its makers, Eastern Color & Chemical Co.

The new product reportedly has proven to be an excellent permanent white pigment for overprinting on dyed background goods inasmuch as it is said to contain noncuring binders, finely dispersed titanium dioxide, and permanent nonyellowing whitener.

Used in discharge printing, Eccoblanc W-45-B is said to ensure permanently white, nonyellowing prints. Sharp prints are assured because the thickening agents and viscosity are such that they will not thin or diminish in any way the viscosity of the print paste, it is claimed.

In appearance, the new Eccoblanc W-45-B is a smooth, white, semifirm paste. A signal advantage is said to be stability to indefinite storage.

Generally, the makers note that Eccoblanc W-45-B is completely compatible with most printing systems, including some w/o systems. Full data, including recommendations for application, are available from Eastern Color & Chemical Co, 35 Livingston Street, Providence 4, RI.

●

Monsanto Expands Surfactant Line

Monsanto Chemical Company's Inorganic Chemicals Division has announced the expansion of its line of nonionic surface-active agents with the addition of alkyl phenol-based ethylene oxide condensates.

Monsanto already markets alcohol- and mercaptan-based nonionics and for years has been a major producer of tall oil-ethylene oxide condensates.

Interim production of the dodecyl phenol and nonyl phenol-based products is already underway at the company's Kearny, NJ, and Everett, Mass, plants. Commercial production is scheduled for this month at Kearny and Trenton, Mich.

The new products, which will be marketed as a part of the company's Sterox line, are based on research conducted over a period of several years covering the technology involved in their manufacture and use. They reportedly offer several combinations of properties not previously available to the industry.

Lanafast Premetallized Dyes

Althouse Chemical Co, Reading, Pa, has announced the development of a new line of dyestuffs, following nearly three years of research and development by Althouse chemists.

The new Lanafast dyestuffs are said to be applicable to wool, nylon, Creslan, Zefran and Acrilan fabrics as well as mixtures and combinations of these fibers. Exhaustive tests reportedly have proven these dyes to have superior light- and wetfastness properties. The Lanafasts are claimed to be especially suitable for upholstery, carpets, mens and ladies suitings and sportswear fabrics over a wide range of fashion shades.

Tested in the Weather-Ometer, they are said to have met all fastness specifications for upholstery and carpeting fabrics. Fade-Ometer tests show a 7-8 rating with no break in shade, and excellent washing and fulling fastness is experienced using these premetallized dyes, it is claimed.

Lanafast premetallized dyes may be used in conjunction with each other for the production of compound shades since they have similar dyeing and fastness properties. Application of these dyestuffs is carried out with standard application procedures.

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2 SILICATES OF EXCEPTIONAL PURITY*

For continuous or batch bleaching with hydrogen or sodium peroxide, or hypochlorite-peroxide.

- **Purified to prevent precipitation.**
- **Purified to prevent wasteful decomposition of bleach.**
- **Prepared for individual mill needs (Starso's higher alkalinity lowers consumption of NaOH.)**

	Weight Ratio $\%Na_2O:\%SiO_2$	$\%Na_2O$	$\%SiO_2$	°Be. at 68°F.
STAR	1:2.50	10.6	26.5	42.0
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*Processed to minimize impurities such as iron and titanium to avoid catalytic decomposition of peroxide.

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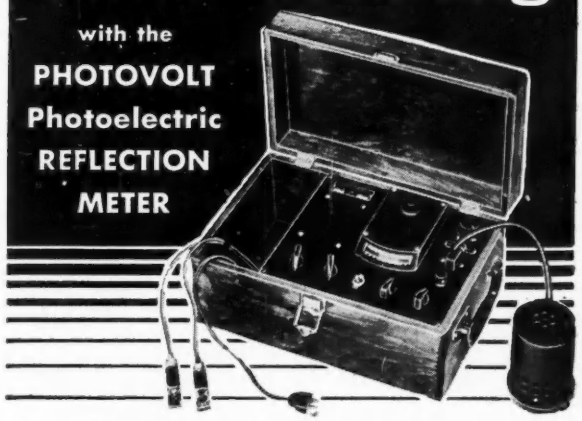

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BOOK REVIEW

Physical Methods of Investigating Textiles

Edited by R Meredith and J W S Hearle
Published by Interscience Publishers, Inc, 250 Fifth Ave, New York 1, NY (1959)
pp 412 + pp 26 index

The editors of this book, R Meredith and J W S Hearle, are members of the faculties of the Royal College of Science and Technology of Glasgow, and the Manchester College of Science and Technology, respectively. The other contributors, all chosen for their specialized knowledge, include D Tabor, G King, W E Morton, D R Holmes, J C Guthrie, R C Faust, J A Chapman, C G Cannon, R Barer and S H Anderson.

This volume resulted from the success of a series of lectures, held at the Manchester College of Science and Technology in 1957, that was conducted to describe the modern experimental methods of determining the structure and physical properties of textiles. The emphasis throughout this volume is on the newer techniques, many traditional methods being omitted. The applications of physics to textile technology now include the study of the fine structure of fibers, using methods such as x-ray diffraction, electron microscopy and infrared spectroscopy. This text also includes a chapter on the "Applications of Nuclear Physics". Each section is accompanied by an extensive, up-to-date reference list. The book is well illustrated, including many photographs, and should prove to be an indispensible desk reference for the textile technologist and manufacturer.———RK.

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Modern Chemical Processes—Vol V

Published by Reinhold Publishing Corp, New York, NY
May 20, 1958
pp 154; 8 x 11; $5.00

This is the fifth volume of a series describing American chemical technology in pertinent detail. This volume describes the chemical-manufacturing plants involved in the production of phosphorous, petrochemicals from natural gas, tantulum, granulated fertilizers, uranium from gold wastes, interesterified lard, boron chemicals, cottonseed oil, potassium borohydride, sulfuric acid from anhydrite, mercaptar-free gasoline by the dualayer process. Each section is accompanied by a reference list and an index appears at the end of the volume. Flow diagrams, pictures, and charts provide ample illustrations.———RK

General Calendar

AMERICAN ASSOCIATION FOR TEXTILE TECHNOLOGY
Mar 2, Apr 6, May 4, June 1 (Della Robbia Room, Hotel Vanderbilt, New York, NY)

AMERICAN COTTON MANUFACTURERS INSTITUTE
April 7-9 (11th Annual Meeting, Americana Hotel, Bal Harbour, Miami Beach, Fla); Mar 23-25 (12th Annual Meeting, Fontainebleau Hotel, Miami Beach, Fla)

AMERICAN SOCIETY FOR QUALITY CONTROL, TEXTILE DIVISION
Feb 18-19 (10th Annual Conference, Clemson House, Clemson, SC)

AMERICAN TEXTILE MACHINERY ASSOCIATION
May 23-27, 1960 (American Textile Machinery Exhibition, Auditorium, Atlantic City, NJ)

FEDERATION OF BRITISH INDUSTRIES
June 10-26 (British Exhibition of Industry, Technology, Science and Culture—Coliseum, New York, NY)

INTER-SOCIETY COLOR COUNCIL
April 11-12, 1960 (29th Annual Meeting—Philadelphia Museum College of Art, Philadelphia, Pa)

NATIONAL COTTON COUNCIL
Feb 8-9 (Annual Convention, Dallas, Texas); Sept 28-29 (9th Chemical Finishing Conference, Statler Hotel, Washington, DC)

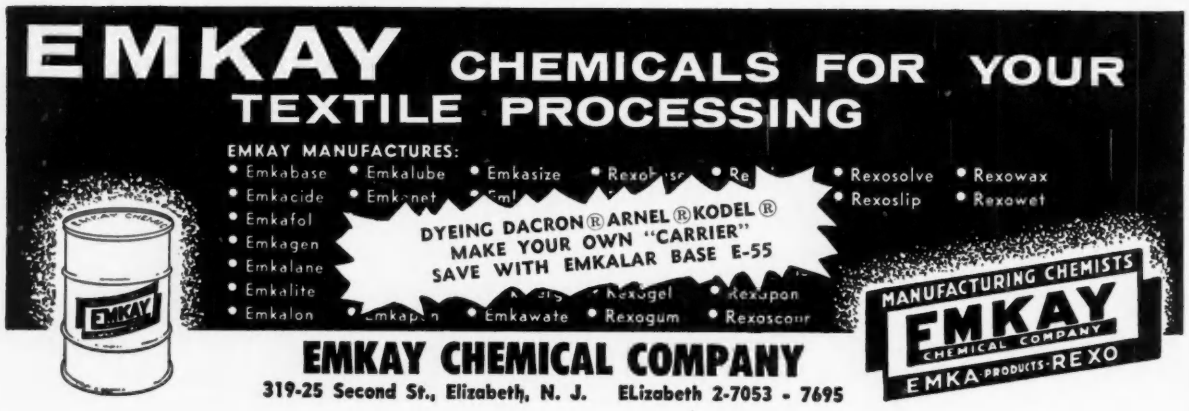
PHI PSI FRATERNITY
April 28-30 (57th Annual Convention, Hotel Roosevelt, New York, NY)

SOUTHERN TEXTILE EXPOSITION
Oct 3-7 (Greenville, SC)

SYNTHETIC ORGANIC CHEMICAL MANUFACTURERS ASSOCIATION
Mar 15, Apr 12 (Luncheon meetings, Hotel Roosevelt, New York, NY); May 11-13 (Spring Outing, Shawnee Inn, Shawnee-on-Delaware, Pa); June 14, Sept 13, Oct 11, Dec 8 (Hotel Roosevelt, New York, NY)

TECHNICAL ASSOCIATION OF THE PULP AND PAPER INDUSTRY, DELAWARE VALLEY SECTION
Mar 26 (3rd Philadelphia Conference of the Graphic Arts Industry—Benjamin Franklin Hotel, Philadelphia, Pa)

TEXTILE RESEARCH INSTITUTE
March 24-25, 1960 (30th Annual Meeting, Hotel Commodore, New York, NY)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